

PRETREATMENT OF BITUMINOUS COALS FOR  
PRESSURE GASIFICATIONGeorge P. Curran, Bedrich Pasek,  
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Eastern steam coals become fluid when heated through the temperature range of 700 to 850°F, and therefore require pretreatment to prevent caking at gasification conditions. Pretreatment via preoxidation has been studied for processes under development by Consol, IGT and the USBM.

Consolidation Coal Co. has carried out work for the past several years on the development of a fluidized bed gasification process for the production of low-sulfur fuel gas from high-sulfur caking bituminous coals. Progress reports have been published<sup>(1,2,3,4)</sup> from time to time giving the status of the development.

The process involves gasification with air and steam in a fluidized bed of the pretreated coal at temperatures in the range of 1700-1750°F and at pressures of about 15 atmospheres. The pretreatment method studies most intensively is that of preoxidation.

Two criteria are used to evaluate the effect that preoxidation requirements have on economics. The first is the percent preoxidation required as compared with the adiabatic quantity, i.e., the amount of oxidation by heat balance to sustain the reaction at the desired temperature. With percent preoxidation defined as pounds O<sub>2</sub> reacted/100 pound dry coal fed, this relationship is illustrated in Figure 1. If the percent preoxidation exceeds the adiabatic level, the reactor becomes more complicated because of the need for cooling by indirect heat transfer and the heat released cannot be utilized efficiently.

The other criterion for preoxidation relates to the fluidization behavior of the preoxidized coal product. In order to operate the gasifier at a practical throughput, it is necessary to use a relatively coarse feed, and for the preoxidized coals to have a relatively high particle density. These properties are required in order to permit operation at reasonable gas velocities without excessive entrainment and to maintain the required bed inventory to satisfy the demands of the gasification kinetics.

The difficulties involved in meeting these criteria when preoxidizing Pittsburgh Seam coal at pressure have led to exploratory work on a pretreatment process in which the fluid coal is smeared out over seed char particles to produce a dense noncaking gasifier feedstock. The technique has been designated the "Seeded Coal Process."

The "Seeded Coal Process" in principle would actually utilize the natural fluidity of the coal. In the process visualized, char would be circulated at a high rate by means of a lift gas through a draft tube immersed in a normal fluidized bed. Coal and fine size seed char would be fed into the draft tube. The external fluid bed would be maintained at 1000-1400°F either by injection of air or hot fluidizing gas from a gasification step.

The coal would melt, smear out over the surfaces of the seed char and external bed material, and then solidify on completion of pyrolysis.

BACKGROUNDPreoxidation

Pretreatment via preoxidation has been studied by many investigators as a means of rendering caking coals operable for subsequent processing. The degree and severity of pretreatment required is a function of the type of processing the coal will be subjected to and the conditions under which the process is operated, i.e., temperature, pressure and gas composition, as well as the particular type of processing equipment used.

The following are some specific examples of the effect of operating conditions on fluid bed processing. First, it may be noted that more severe pretreatment is required for processes that operate under pressure as compared to those that operate at atmospheric pressure.

The effect of increasing total pressure is illustrated by the two experimental observations outlined below. Ordinarily, Pittsburgh Seam coal cannot be fed directly into a gasifier. However, a highly caking Pittsburgh Seam coal was successfully processed in 1949 in an atmospheric pressure one ton per hour fluid bed gasification unit without any pretreatment<sup>(5)</sup> whatsoever. The above admittedly was accomplished at a relatively low coal throughput rate of 25 lb/hr-ft<sup>2</sup>, but the effect of higher rates was not explored.

The other observation was, that in processing noncaking subbituminous coals at 20 atmospheres pressure in the hydrodevolatilizer of the CO<sub>2</sub> Acceptor Process, agglomeration of the bed solids occurred unless the coal feed was pretreated by mild preoxidation.<sup>(6)</sup> The need for pretreatment here may be affected also by the presence of a substantial partial pressure of H<sub>2</sub>, i.e., 5.5 atmospheres.

The work on the development of the Synthane Process at the USBM<sup>(7)</sup> illustrates the fact that successful operation of a pressurized fluidized bed gasification process with bituminous coal requires that the feed be pretreated by preoxidation.

Work at IGT on the development of the Hydrogasification Process,<sup>(8,9,10)</sup> again illustrates the need for severe pretreatment to establish operability in the fluidized bed processes operated at high total pressures, i.e., ca. 1000 psig. The high partial pressure of hydrogen in the hydrogasifier may also intensify the need for pretreatment.

A large experimental effort was carried out in the laboratories of Consolidation Coal in the 1950's to define the minimum severity of pretreatment via preoxidation required to establish operability in a subsequent atmospheric pressure fluidized bed carbonizer operated at 950°F. The aforementioned work has not been published, but the salient conclusions are given here.

The severity of pretreatment may be minimized by maximizing the amount of associated thermal treatment, i.e., pretreatment temperature and residence time. There is, however, for each specific coal a maximum pretreatment temperature that may be used above which the pretreatment process itself becomes inoperable. For highly caking Pittsburgh Seam coals, the "optimum" pretreatment temperature is in the range of 750-800°F.

The procedure finally adopted<sup>(11)</sup> was a mild preoxidation at temperatures below the plastic zone, i.e., <600°F, followed by final preoxidation at temperatures within the plastic zone, i.e., at 725-800°F. The total preoxidation required for the LTC process was in the range of 5-8 wt %, and increased with increasing fluidity of the coal being processed.

The use of preoxidized coal for fluidized bed<sup>(12)</sup> reduction of calcium sulfate at temperatures of 1825-1875°F but at low pressures, i.e., 8 psig, has also been described. In this instance, 5.3 wt % preoxidation of the feed coal at 8 psig and 700°F was sufficient to prevent defluidization due to coking. The same highly fluid Pittsburgh Seam coal, Ireland Mine, and the same particle size (28 x 100) was used as in the work to be discussed here.

Forney,<sup>(13)</sup> et al. studied the effect of the variables on ease of pretreatment of caking coals in a fluidized bed. They concluded, in accord with other investigators, that higher temperatures, increased residence time and decreased particle size all decreased the amount of preoxidation required to make the feed coal noncaking.

The development of the Synthane Process for gasification of bituminous coal most closely parallels the work reported here as far as the need for pretreatment is concerned. The small-scale pilot work<sup>(14)</sup> used a free fall, dilute-phase preoxidizer to pretreat the coal. With very fine Pittsburgh Seam coal, i.e., 70% through 200 mesh, less than 8 wt % preoxidation was sufficient to decake the feed coal.

The large Synthane pilot plant now under construction will, however, incorporate a more complex two-stage pretreatment procedure. This is considered necessary since the coarser feed coal size (-14 M x 0) to be used necessitates more severe pretreatment. The first stage is a fluid bed preoxidizer operated at 40 atm and 750°F, followed by a second precarbonization stage which is conducted by free fall of the preoxidized coal against the product gases through the extended free board zone of the gasifier. With this system approximately 8 wt % preoxidation is considered to be adequate to achieve operability with a Pittsburgh Seam coal.<sup>(15)</sup>

#### Seeded Coal Process

A demonstration of the technique which is to be applied to the "Seeded Coal Process" was successfully carried out in the low-temperature carbonization section of the CSF Coal Liquefaction Pilot Plant at Cresap, West Virginia.<sup>(16)</sup> The feed material, in this instance, was somewhat different and constituted the underflow from the hydroclone separation of the extraction effluent. Coal extract in this case was used instead of the fluid coal and the extraction residue was used instead of the seed char. Other differences were that the mixture was sprayed into the draft tube as a slurry and operating temperatures and pressures were lower, i.e., 825-925°F and approximately 4 psig, respectively.

In this particular installation, a 36" I.D. carbonizer was employed in which there was installed a 6" I.D. x 11' high draft tube. Solids were circulated through the draft tube by injection of about 3500-4500 SCFH of lift gas into the bottom of the tube. The feed was sprayed into the circulated char stream within the draft tube by means of a nozzle 3 feet above the lift gas injection point.

Solids circulation rates of the order of 100,000 lb/hr were achieved in this device, while complete operability and product size control was maintained with extract feed rates up to 200 lb/hr. The ratio of extraction residue solids-to-extract was in the range of about 1.5/1 to 3/1. The above throughput rates do not necessarily represent the capacity of the system since higher extract feed rates were not available and consequently were not tested.

The above results led to an attempt to apply the same system to coal even though coal is a less fluid material than extract and the operating conditions, particularly the pressure required, are more severe.

EQUIPMENT AND PROCEDUREBench-Scale Unit

A flow diagram of the experimental preoxidation unit is shown in Figure 2. The reactor is shown with the draft tube in place as for seeded coal tests. Preoxidations were generally carried out without the draft tube unless otherwise stated. Coal was fed at a known rate through a rotary feeder into a pneumatic lift line which conveyed it into the bottom of the reactor. Pretreated coal overflowed a weir and was collected in a separate product receiver.

For the seeded coal and draft tube runs, several reactor configurations were employed as shown in Figures 3 and 4. Another feeder was used for the seed char and an additional line was installed for accelerating gas to the draft tube. The reactor was 4" I.D. with an active bed height of 40". The reactor internals were constructed of type 310 stainless steel.

Fluidizing gas was metered through rotameters. It entered the top of the reactor through a preheat coil into the bottom of the bed where it reversed direction and fluidized the bed. Preoxidizer outlet gas generally contained some heavy tar and pitch which would rapidly plug the solids filters and the outlet piping. This was alleviated by installing two parallel tar receivers to collect the heavy material.

After passing through the coolers, the gas was depressured and analyzed. The analytical train consisted of a continuous paramagnetic oxygen analyzer and infrared SO<sub>2</sub>, CO<sub>2</sub> and CO analyzers. In addition, the gas was selectively sampled for intensive chromatographic analysis.

Laboratory Test for Gasifier Operability

To judge the likelihood of caking in the gasifier, a laboratory test was developed which consisted of fluidizing a sample in a quartz tube reactor with nitrogen, and then immersing the reactor into a fluidized sand bath furnace for three minutes. The temperature in the reactor reached 1500°F within two minutes. If no agglomeration occurred, the material would surely be operable in the gasifier. Samples which showed slight agglomeration might be operable in the gasifier since the coal is fed in with a stream of air.

The FSI method has been used by previous investigators<sup>(13)</sup> to test for decaking for subsequent gasification. We have found this method unsuited to our system since samples with FSI values as low as 1 were inoperable in the gasifier.

Similarly, the Gieseler plastometer was also used for the same purpose. The maximum fluidity of an operable feedstock was below the level that can be quantitatively measured, i.e., <0.1 DDPM.

EXPERIMENTAL RESULTSBench-Scale Preoxidation of Illinois No. 6 Coal

Three runs were made with Illinois No. 6 coal from Consol's Hillsboro Mine. This coal can be classified as weakly caking. Tests with a standard Gieseler plastometer showed a maximum fluidity of only 2.8 DDPM.

The conditions of the runs are summarized below:

System Pressure: 15 atm (206 psig)

<u>Run Number</u>	<u>5P1</u>	<u>5P2</u>	<u>6P</u>
Temperature, °F	750	800	810
Inlet O <sub>2</sub> Partial Pressure, atm.	0.31	0.32	0.25
Fluidizing Velocity, ft/sec (top of bed)	0.29	0.29	0.29
Percent Preoxidation	11.9	11.5	8.7

Run data, properties of the preoxidized products, material balances and distribution of oxygen in the products are given in Tables 1 through 4.

The first runs were made with the level of preoxidation conservatively above the adiabatic level, 11.7%.

Upon completion of the 750°F portion of Run 5P1, the temperature was raised to 800°F via the electrical preheaters in order to obtain a more highly devolatilized product. Both runs were completely operable.

In Run 6P, the amount of preoxidation was reduced to approach the adiabatic level. The temperature was increased slightly to 810°F, compared to 800°F in Run 5P2.

In Run 6P, the pressure drop across the fluidized bed increased continuously. Microscopic examination of the chemical analysis of the final bed material drained from the reactor showed that ash particles had accumulated in the bed during the run. Ash and pyritic sulfur balances on the product coal and bed material showed that 10.55% of the dry feed coal had accumulated in the bed compared with 10.29% as obtained by difference in the overall weight balance, as shown in Table 3. A check of the chart records for Run 5P2 also showed an increase in pressure drop across the bed, but to a lesser extent than in Run 6P. In Table 3 the accumulations in Runs 5P1 and 5P2 were obtained by differences in the overall weight balances, in view of the good agreement between the measured and difference values shown in Run 6P. The nominal retention times shown in Table 1 are corrected for the presence of accumulated ash.

The preoxidized coal products from Runs 5P2 and 6P were fed to the bench-scale gasifier operated at simulated process conditions shown in Table 5. Both gasification tests were completely operable with no traces of agglomeration. All further work concentrated on the Ireland Mine feedstock.

#### Bench-Scale Preoxidation of Pittsburgh Seam Coal

The coal used for these tests was from Consol's Ireland Mine. To the best of our knowledge, this is the most fluid of the Pittsburgh Seam coals. The maximum fluidity is >37,000 DDPM, i.e., greater than can be measured in the standard Gieseler plastometer. Successful preoxidation of Ireland Mine coal would ensure applicability of the process to any other coal.

For the initial experimental program, a severe level of preoxidation was chosen deliberately with the intent of suppressing completely any tendency toward caking at gasification conditions. A long nominal retention time, 60 minutes, was chosen to ensure adequate exposure to O<sub>2</sub> for those particles at the low end of the retention time distribution which is characteristic of a continuously fed fluidized bed. With these constraints, the inlet O<sub>2</sub> partial pressure fell in the range of 0.4-0.5 atm.

#### Run 1P

Run data and properties of the preoxidized coal products are listed in Tables 6 and 7. The run proceeded without apparent difficulty, except for a disturbingly large temperature gradient across the fluidized bed, until the desired amount of product was

fed. During the run, the temperature at the bottom typically was 580°F and the profile showed an increase to 700°F at about 25" above the bottom with constant temperature to the top of the bed at 40".

On disassembly of the reactor it was found to be partially choked with large chunks of agglomerated coal. The presence of agglomerates distorted the normal mixing pattern of the fluidized bed and also decreased the nominal residence time of the coal particles by an unknown amount. However, all the O<sub>2</sub> had been consumed, i.e., 20% preoxidation had been achieved.

#### Run 2P1

Since coke had formed at 700°F in Run 1P, the next preoxidation run was made at 600°F to guarantee a preoxidized coal feedstock with a known retention time which could be fed to the gasifier.

In Run 2P1, a large temperature gradient developed soon after coal feeding was started. After two hours, the temperature increased suddenly to 800°F at which time the run was ended. Upon disassembly, there was no evidence of agglomeration, yet the temperature effects indicated that agglomerates had formed.

Laboratory tests showed that although the coal would not agglomerate at atmospheric pressure at 600-650°F, agglomeration could occur at pressure. This behavior was attributed to small amounts of waxy and resinous material which are present in nearly all bituminous coals. This material melts at relatively low temperatures and evaporates at atmospheric pressure before agglomerates can form. At elevated pressure, evaporation is hindered sufficiently so that the particles can stick to each other. The agglomerates so formed must have been very fragile and were broken up during manipulation of the reactor on disassembly after Run 2P1. In Run 1P the start-up procedure involved heating the coal through the 600-650°F range and agglomerates probably formed at that time.

#### Run 2P2

The start-up procedure was changed to ensure that the bed temperature always remained above 725°F. The run was carried out successfully at 750°F. Run data, some properties of the preoxidized coal product, the distribution of reacted O<sub>2</sub>, and a material balance are presented in Tables 6 through 9.

Both Runs 1P and 2P2 products were fed to the gasifier at conditions described in Table 5. The Run 1P material melted and coal feeding had to be stopped after 13 minutes. The Run 2P2 material produced numerous small agglomerates.

It was apparent that, at the pressure used in the process, even 20% preoxidation was ineffective in preventing caking in the gasifier. Pretreatment by preoxidation involves dehydrogenation and devolatilization. Elevated system pressure inhibits these processes while increased preoxidation and temperature enhance these same processes. Since a higher preoxidation temperature can compensate for the suppressing effect of elevated pressure on dehydrogenation and devolatilization, preoxidation runs at 800°F were scheduled.

#### Runs 3P and 3P1

Runs 3P and 3P1 were made at 800°F. In both cases, soon after 800°F was reached in the bed, there was evidence of poor mixing or blockages in the bed. The beds were found to contain large amounts of agglomerates.

Runs 4P and 4P1

To establish a benchmark as to the severity of preoxidation for Ireland Mine coal which would assuredly provide an operable feedstock for the gasifier, the Run 2P2 product was subjected to a second stage of preoxidation in Run 4P at 750°F in which an additional 10% preoxidation occurred. Run data, some properties of the preoxidized coal product, the distribution of reacted O<sub>2</sub>, and a material balance are presented in Tables 6 through 9.

Run 4P product was fed to the gasifier at conditions shown in Table 5. There was no evidence of agglomeration or ash slagging in the gasifier.

In order to obtain a gasifier feedstock which was more highly devolatilized, at the end of the 750°F operating period of Run 4P, the bed temperature was raised to 800°F while holding all input flows constant.

Increased particle swelling was apparent immediately. After about one hour at 800°F, the very low density material would not flow reliably through the overflow weir, and the run was shutdown. Inspection of the vessel after disassembly showed that no caking or agglomeration had occurred.

However, such a low density material, particle density less than 30 lb/ft<sup>3</sup>, would be impractical as a gasifier feedstock from the standpoint of maintaining an adequate carbon inventory and gas throughput.

Reference to Tables 2 and 7 shows that the density of the preoxidized coal product increased with decreasing particle size. This is illustrated in Figure 5 using the data from Runs 1P and 2P2. Results of Gieseler plastometer tests on the various fractions showed that the smaller particles also displayed less fluidity. It was concluded that a smaller size consist would be more operable in the gasifier than a larger particle which had experienced the same degree of preoxidation.

Staged Preoxidation Run

A series of runs were then made in which a finer size consist, nominally 48 x 150 mesh, of the raw coal feed was used in conjunction with two stages of preoxidation. From prior work both expedients are in the direction of reducing the excessive preoxidation required in single-stage treatment of relatively coarse coal.

a. First Stage - Run PR1

Programmed conditions called for the adiabatic level of 6.3% preoxidation. The actual coal feed rate was somewhat higher than the programmed value, with the result that 5.7% preoxidation was achieved. The run was completely operable and scheduled shutdown was made after making 143 pounds of steady-state product.

b. Second Stage at 775°F - Run PR1B

After steady-state conditions at 750°F had been reached in Run PR1A in which complete operability was demonstrated, the bed temperature was raised to 775°F for Run PR1B. The preoxidation level was increased slightly to compensate for lower level achieved in the first stage run such that the total level for both stages was 11.3% preoxidation, which is 50% greater than the adiabatic level at 775°F. This run was also completely operable with no caking or agglomeration occurring.

The Run PR1 product and all the air were fed into the configuration D draft tube shown in Figure 4. In a bench-scale preoxidation run, the draft tube probably makes little contribution to the process. In the tube, the incoming first-stage coal product and air were immediately diluted with a large amount of external bed material.

The draft tube was used to assure a smooth transition from start-up conditions involving an initial bed of inert char and to prevent localized hot spots caused by the highly exothermic preoxidation reactions. Axial traverses across the entire bed showed that the temperature was constant to within 4°F.

c. Second Stage Preoxidation Attempt at 800°F

In an effort to obtain a more highly devolatilized, and therefore a more thoroughly decaked product, the bed temperature was raised to 800°F via the electrical heaters at the end of the material balance period of Run PR1B. After about one-half hour, the pressure drop across the bed had increased and a large temperature gradient had developed. Inspection of the reactor after shut down showed that it was full of large chunks of agglomerates.

Detailed results of Runs PR1 and PR1B are given in Tables 6 through 9. Inspections of the preoxidized coal product from Run PR1B show that the material was considerably swollen and had a particle density of 49 lb/ft<sup>3</sup>, compared with the raw coal value of 82 lb/ft<sup>3</sup>. This result was not anticipated because our prior work had indicated that the smaller size fractions of the preoxidized coal products had suffered relatively little swelling. Some comparisons are shown below with data from two runs made during the earlier work.

<u>Run Number</u>	<u>1P</u>	<u>2P2</u>	<u>PR1B</u>
Raw Coal Size Consist	← 24 x 100 M →		48 x 150 M
Temperature, °F	700	750	775
Percent Preoxidation	19.5	18.6	11.3
<u>Particle Density, lb/ft<sup>3</sup></u>			
Mean of Entire Product	61.6	53.1	48.6
65 x 100 mesh Fraction	76.8	73.1	54.4

The lower density of 65 x 100 mesh product produced in PR1B as compared with previous work may be because of the lower level of preoxidation and/or the higher temperature used.

The laboratory shock-heating test showed that the Run PR1B product would have been inoperable at gasifier conditions. The amount of agglomerates formed in the test was greater than that for the Run 2P2 product which actually was fed to the gasifier at process conditions and which was shown to be inoperable.

DISCUSSION OF PREOXIDATION RESULTS

The results obtained in this study are qualitatively in agreement with the results of previous investigators as discussed above. Particularly noteworthy is the adverse effect of the process pressure on the severity of pretreatment required to establish operability.<sup>(5,6,8,9,10,12)</sup>

Staged preoxidation with a rising temperature regime between stages also appears moderately helpful in reducing the severity of pretreatment as shown by others.<sup>(7,11)</sup> However, as noted above, for each coal, and each given size consist, there is a maximum pretreatment temperature at which the first pretreatment step itself remains operable. For the highly fluid Pittsburgh Seam coals, the maximum first-stage pretreater temperature at 15 atm pressure for 28 x 100 mesh feed is about 750°F.

Fine coal requires less severe pretreatment to achieve the same degree of decaking, which again is in agreement with other investigators.<sup>(13)</sup>

The level of preoxidation required to achieve operability with Pittsburgh Seam coals, as reported here, is substantially in excess of that indicated for the Synthane Process.<sup>(15)</sup> A possible explanation is the added pretreatment received by

the coal in the Synthane Process by precarbonization in the free falling zone above the bed. Such added pretreatment is avoided in the Consol process to eliminate complications due to tar formation. The same result could possibly be achieved in the Consol process by addition of an extra preoxidation stage in the form of a transfer line reactor. This would be done by conveying the pretreated coal with air from the preoxidizer to the gasifier.

Another factor which is brought out in this study is that more severe pretreatment is required to decake the coal when the pretreatment step itself is conducted under pressure. This is illustrated by the results shown in Table 10, where the caking properties of Ireland Mine coal preoxidized at 1 and 15 atmospheres pressure are compared. The data of Table 10 also again illustrate the favorable effect of higher pretreatment temperatures in decaking, i.e., comparison of Runs 1P and 2P2.

The problem of pretreatment is also compounded by the need, as discussed above, to process a relatively coarse feed without a concomitant large decrease in particle density.

Noteworthy also is the excellent kinetics of the preoxidation step. Substantially complete oxygen consumption was observed in all of the preoxidation runs reported here. Supporting laboratory data on preoxidation kinetics are also given in reference (2). These data<sup>(2)</sup> also point out an interesting finding that higher oxygen partial pressures cause a higher degree of decaking at the same preoxidation level.

#### PRETREATMENT VIA "SEEDED COAL PROCESS"

The principle of the preoxidation method of pretreatment is to convert the coal to a more rigid structure via oxidation such that the fluidity is severely reduced when the coal undergoes pyrolysis. The Seeded Coal Process would operate on just the reverse principle and actually utilize the natural fluidity of the coal. Unfortunately, we were severely handicapped by the small scale of the equipment available since the draft tube principle had to be adapted to the existing 4-inch diameter vessel.

The potential advantages of the process are that it will supply a feedstock that is assuredly operable with respect to agglomeration at gasifier conditions. In addition, it has the capability to produce a dense, closely sized feedstock substantially free of fines. This will permit a higher gasifier throughput than otherwise.

A series of exploratory tests were carried out with the configurations A, B and C as indicated in Figure 3. Best results were obtained with configuration C, but even here two basic deficiencies were noted. From the appearance of the agglomerates obtained, it was apparent that insufficient mixing was being achieved in the draft tube between the injected coal and the circulating char. Part of the difficulty is associated with the small scale of the equipment, since calculations show that the particle Reynolds number in the draft tube is barely above the Stokes Law range. Also, it was apparent that most of the fluidizing gas was bypassing the main bed in favor of the draft tube. The result was that a fluidized bed was not maintained external to the draft tube.

To overcome these limitations, the configuration C of Figure 3 was modified as follows:

To allow installation of an external baffle which would maintain fluidization of the external bed, the draft tube was raised 2 inches and the inlet lines were lengthened accordingly. An elliptical baffle 3-5/8" x 1-3/4" x 1/16" thick was welded to the accelerating gas line below the mouth of the tube at a slope of 60° from the horizontal. To help promote mixing a conical baffle was installed inside the tube with the apex of the cone positioned 1/2 inch above the end of the coal inlet tube. The configuration D obtained is shown in Figure 4.

Tests with an inert bed of 48 x 100 mesh char at 1500°F and 15 atm. system pressure showed that the external baffle was effective. The solids circulation rate upward through the tube was measured by substituting a known amount of air for some of the N<sub>2</sub> entering the solids feed line. From the measured temperature rise, the solids flow rate was calculated as 900 lb/hr by heat balance. Calculations involving the pneumatic transfer line<sup>(17)</sup> model devised in the course of development of the CO<sub>2</sub> acceptor project showed that without the external baffle about 270 of the 340 SCFH of N<sub>2</sub> fed to the bottom of the external bed had entered the draft tube whereas with the baffle, the flow was reduced to about 60 SCFH.

Seven tests were made with the modified draft tube, using an external bed of 48 x 100 mesh char at 15 atm. system pressure. Common conditions for the runs are listed below:

Ireland Mine coal: sized to 100 x 200 mesh  
 Coal Feed Rate: 2.0 lb/hr  
 Duration of Feeding: 3.3 minutes  
 Air to Coal Feed Line - Equivalent to 100% of adiabatic  
 preoxidation level at the temperature used.

Gas Flows, SCFH

Air + N <sub>2</sub> to Coal Feed Line	65
N <sub>2</sub> to Accelerating Line	85
N <sub>2</sub> to Bottom of External Bed	340

Tests were made at temperatures from 900 to 1500°F, in 100° increments. Temperature limits of operability were established as follows: (1) at 900°F little or no smearing occurs as was shown by presence of coal-derived material in the form of hollow spheres in the bed after the run, and (2) at 1500°F, caking occurred in the draft tube.

The products from the runs at 1000 to 1400°F all showed more uniform smearing than in any of the previous runs without the internal baffle. At the end of each run, the system was depressured and the bed was drained by removing the coal inlet line. The hot bed material was quenched rapidly by contact with dry ice in the catchpot. The entire bed material then was screened at 28 and 48 mesh. A characteristic of all the run products is that all contained some +48 mesh agglomerates which were external bed particles cemented together by a thin film of coal-derived material. No agglomerates larger than 28 mesh were found. The fewest agglomerates occurred at 1300°F, indicating that this may be the optimum temperature with respect to uniformity of smearing. The amounts of +48 mesh agglomerates which formed are listed below:

Temperature, °F	+48 Mesh Agglomerates/Wt. % of Bed Inventory
1000	18.0
1100	16.0
1200	15.5
1300	8.0
1400	10.7

The particle density, measured in mercury, for the +48 mesh agglomerates formed at 1300°F had a high value of 85 lb/ft<sup>3</sup>.

An attempt was made to run for a prolonged period at 1300°F and 15 atm. system pressure to determine the size distribution of the "equilibrium" product. To simulate the seed char in the commercial embodiment (fines from the internal cyclones in the gasifier) an initial external bed of -100 mesh precarbonized char was established.

Then, 100 x 200 mesh Ireland Mine coal and additional -100 mesh char were fed to the draft tube at rates of 2 and 4 lb/hr, respectively. The fine char contained a considerable amount of -325 mesh material which was elutriated from the reactor. The outlet piping system of the present equipment was not designed to handle large amounts of solids. The run had to be terminated after 35 minutes of feeding coal because the outlet system began to plug. Thus, an equilibrium bed was not established. However, analysis of the bed showed that it contained 50 wt % of +100 mesh agglomerates, with a top size of 24 mesh.

The high particle density achieved is favorable, in that "smearing" of liquid coal over the seed particles apparently is occurring as desired.

The small size of the existing equipment precludes any further meaningful studies of the Seeded Coal Process. The radial clearance between the inlet line and the wall of the draft tube is only 0.15 inch. The mouth of the tube eventually would become choked by the larger agglomerates which inevitably would be formed.

The results of the exploratory studies strongly indicate that future studies should be made.

Several essential factors are required to achieve success in such an operation. Intensive mixing in the draft tube is required to achieve smearing of the "liquid" coal over both the seed and recirculating char. A sufficient residence time in one pass through the unit of the recirculating burden is needed to complete the "drying out" or carbonization of the coal. Finally, the draft tube must be large enough to handle without choking, the largest size particles made in the process. All these factors point to a need for a larger unit in which the draft tube would be at least 2 inches in diameter as opposed to the present 0.680 inch. Such a unit, of course, would have a much higher capacity for coal feed which would lie approximately in the range of 30-300 lb/hr.

#### CONCLUSIONS

1. Encouraging results have been obtained in an exploratory study of the Seeded Coal Process. Future studies should be made with a larger reactor unit in which the draft tube diameter would be at least 2 inches (vs. 0.680 inch at present). The potential advantages of the process are that it will supply a feedstock which assuredly is operable with respect to caking/agglomeration at gasifier conditions, and that it can produce a dense, closely sized feedstock substantially free of fines which will allow a high gasifier throughput.
2. Preoxidation is an acceptable pretreatment technique for Illinois No.6 coals. A feedstock was produced at adiabatic preoxidizing conditions which was fed to a pressure gasifier with complete freedom from agglomeration.
3. Successful pretreatment within the framework of an adiabatic preoxidation process was not achieved with highly fluid Pittsburgh Seam coals using a size consist sufficiently coarse to be practical for fluidized bed processing when pretreatment was carried out under full system pressure. The minimum preoxidation level found is between 19 to 28 wt %. A three-stage process in which the third stage preoxidation is carried out in a transfer line reactor is a possibility which has not been explored.

#### Acknowledgment

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TABLE I

Preoxidation Conditions and Results for Illinois No. 6 Coal  
 System Pressure: 15 atmospheres (206 psig)  
 Size Consist: 24 x 100 Tyler Mesh

Run Number	5P1	5P2	6P
Feed Coal	Illinois No. 6		
Temperature, °F	750	800	810
Inlet O <sub>2</sub> Partial Pressure, atm	0.31	0.32	0.25
Fluidizing Velocity (top of bed), ft/sec	0.29	0.29	0.29
Moisture in Coal, Wt %, as fed	6.80	6.80	6.58
<u>Input</u>			
Coal Feed Rate, lb/hr (dry basis)	7.89	8.13	8.71
<u>Lift Gas, SCFH</u>			
Air	54.0	54.0	43.5
N <sub>2</sub>	97	99	110
<u>Fluidizing Gas, SCFH</u>			
Recycle	396	380	382
N <sub>2</sub>	0	0	0
Purges (N <sub>2</sub> ), SCFH to bed	5	5	5
Purges (N <sub>2</sub> ), SCFH above bed	10	9	6
<u>Output</u>			
Exit Gas Rate, SCFH <sup>(1)</sup> (dry basis)	161	164	161
Exit Gas Composition, Mol % (dry basis)			
O <sub>2</sub>	<.01	<.01	<.01
CO <sub>2</sub>	3.03	3.36	2.54
CO	.40	.46	.25
CH <sub>4</sub>	.25	.38	.45
C <sub>2</sub> H <sub>6</sub>	.08	.14	
C <sub>2</sub> H <sub>4</sub>	.01	.02	
C <sub>3</sub> H <sub>8</sub>	.01	.06	(2)
C <sub>3</sub> H <sub>6</sub>	.01	.01	
COS	.02	.05	
SO <sub>2</sub>	.16	.18	.09
H <sub>2</sub> S	<.003	<.003	--
H <sub>2</sub>	<.01	<.01	--
N <sub>2</sub> (by difference)	96.03	95.34	96.39
Flow Rate, SCFH, at top of bed	572	563	569
<u>Water, lb/hr (corrected for coal moisture)</u>			
Condensate	.590	.722	.824
Moisture in Exit Gas	.006	.006	.006
Tar, lb/hr	.0934	.155	.292
Preoxidized Coal, lb/hr	7.20	6.81	6.76
Percent Preoxidation	11.9	11.5	8.7
Nominal Solids Retention Time, min	59	46	39
Duration of Steady-State Period, hr	10.0	6.0	5.5
Total Product, Pounds	72	41	37

(1) Standard conditions are 29.92 in Hg and 70°F.

(2) Not measured. Assumed to be the same as in Run 5P2.

TABLE 2

Properties of Illinois No. 6 Coal and Products

Hillsboro Mine Illinois No. 6 Feed Coal		5P1		5P2		6P	
	(1)	(2)					
Hydrogen, Wt % (dry basis)	4.19	4.18	3.18		3.29		3.44
Carbon	60.44	60.14	61.72		67.02		70.61
Nitrogen	1.17	1.22	1.34		1.44		1.36
Oxygen (by diff.)	8.81	7.67	7.81		7.98		5.80
Sulfur	4.46	4.93	4.07		3.72		3.40
Ash	20.93	21.86	21.88		16.55		15.39
Volatile Matter	37.2	36.4	23.4		21.6		22.0
Pyritic Sulfur	2.20	2.26	1.51		1.11		1.37
Sulfate Sulfur	.07	.30	.33		.27		.07
				Density, lb/ft <sup>3</sup>		Density, lb/ft <sup>3</sup>	
Size, Tyler Mesh	Wt %	Wt %	Wt %		Wt %		Wt %
+24	0	0	0.4		1.4		2.0
24 x 28	9.6	15.8	7.9		14.0		17.0
28 x 35	39.3	41.1	33.3		35.5		34.5
35 x 48	34.2	29.0	34.7		32.0		29.4
48 x 65	15.0	12.7	20.2		15.0		14.3
65 x 100	1.3	1.0	3.0		1.8		2.2
-100	0.6	0.4	0.5		0.4		0.5
Mean Diameter, inch (3)	.0166	.0176	.0158		.0171		.0175
Mean Density, lb/ft <sup>3</sup> (4)	80.0	80.0	61.7		49.8		51.8
Maximum Fluidity, DDPM (5)	2.8	--	< 1		--		--

(1) Batch No. 1, used in Runs 5 P1 and 5P2.  
 (2) Batch No. 2, used in Run 6P.  
 (3) Arithmetic mean.  
 (4) Measured in mercury at 1 atmosphere. Reciprocal mean.  
 (5) Measured in Gieseler plastometer (ASTM Method D-2639-67T).

TABLE 3

Material Balances for Preoxidation Runs - Illinois No. 6 Coal

Basis: 100 lb Dry Coal Fed

<u>Run Number</u>	<u>5P1</u>	<u>5P2</u>	<u>6P</u>
Preoxidized Coal	91.25	83.76	77.60
Overhead Fines	.70	.50	.91
Tar	1.18	1.91	3.35
Carbon in CO + CO <sub>2</sub>	2.16	2.39	1.60
Hydrogen in H <sub>2</sub> O	.84	1.00	1.06
Hydrocarbons (C <sub>1</sub> -C <sub>3</sub> )	.40	.72	.72
Sulfur in SO <sub>2</sub> and COS	.30	.38	.22
Coal Oxygen to Products	.61	2.88	4.25
Accumulation in Bed, Ash and S as FeS <sub>2</sub>	<u>2.56<sup>(1)</sup></u>	<u>6.46<sup>(1)</sup></u>	<u>10.29<sup>(1,2)</sup></u>
Total	100.00	100.00	100.00

(1) By difference.

(2) Ash and sulfur balances on product and bed materials gave 10.55 lb accumulation.

TABLE 4

## Distribution of Oxygen in Products of Preoxidation - Illinois No. 6 Coal

Run Number	5P1		5P2		6P	
	Lb/hr	Percent	Lb/hr	Percent	Lb/hr	Percent
Output						
To Water						
Condensate	.524	53.2	.641	54.7	.732	65.0
Moisture in Gas	.005	0.5	.005	0.4	.005	0.4
To CO <sub>2</sub>	.402	40.8	.456	38.9	.338	30.1
To CO	.027	2.7	.031	2.7	.017	1.5
To SO <sub>2</sub>	.021	2.1	.024	2.1	.012	1.1
To COS	.001	0.1	.003	0.3	.003	0.3
In Tar	.006	0.6	.011	0.9	.018	1.6
On Coal (by diff.)	0	0	0	0	0	0
Total	.986	100.0	1.172	100.0	1.125	100.0
INPUT						
From Coal (by diff.)	.048		.234		.370	
Lb/hr O <sub>2</sub> from Air	.938		.938		.755	
Total	.986		1.172		1.125	

TABLE 5

Typical Gasifier Conditions for  
Testing Preoxidized Coals

Temperature	1700°F
Pressure	15 atm (206 psig)
Fluidizing Velocity	0.34 ft/sec
Feed Rate	4.15-4.85 lb/hr

<u>Compound</u>	<u>Feed, Mole %</u>	<u>Top of Bed, Mole %</u>
H <sub>2</sub> O	23.6	15.3
H <sub>2</sub>	0.0	10.0
CH <sub>4</sub>	0.0	0.8
CO	0.0	12.0
O <sub>2</sub> (from air)	6.8	0.0
CO <sub>2</sub>	11.8	12.8
N <sub>2</sub>	57.8	49.0
Inlet Oxygen Pressure, atm.		1.0

TABLE 6  
 Oxidation Conditions and Results - Pittsburgh Seam Coal  
 System Pressure: 15 atmospheres (206 psig)

Run Number	IP	ZP	IP	FP	IRL	IRLB
Feed Coal	Inland Mine	2PZ Product	2PZ Product	IRL	IRLB	IRL Product
Size Control, Tyler Mesh	24 x 100	50 x 100	48 x 150	875	775	775
Temperature, °F	700	750	750	18	40(*)	40(*)
Inlet O <sub>2</sub> Partial Pressure, atm	.49	.40	.32	0.30	0.30	0.30
Fluidizing Velocity (top of bed), ft/sec	.24	.34	.29	1.83	0.71	0.71
Moisture in Coal, Wt. % as Fed	1.58	1.58	1.03	10.44	5.64	5.64
INLET	Coal Feed Rate, lb/hr (dry basis)	6.5	7.46	9.34	35	38.4
Air	N <sub>2</sub>	73	80	84	170	126
N <sub>2</sub>	Recycle	310	390	380	417	417
Fluidizing Gas, SCFH	Purges (N <sub>2</sub> ), SCFH to bed	0	92	0	0	0
Recycle	Purges (N <sub>2</sub> ), SCFH above bed	5	5	5	5	5
N <sub>2</sub>	Exit Gas Rate, SCFH (dry basis)	15.5	15.5	15.5	7	11
CO <sub>2</sub>	Exit Gas Composition, Mol % (dry basis)	169	256	159	212	159
CO	O <sub>2</sub>	<.02	<.02	<.02	.14	<.01
C <sub>2</sub> H <sub>4</sub>	CO <sub>2</sub>	3.53	2.56	3.33	.76	.89
C <sub>2</sub> H <sub>6</sub>	CO	1.20	.85	.89	.80	.44
C <sub>3</sub> H <sub>8</sub>	CH <sub>4</sub>	1.82	.82	.71	.03	.16
C <sub>4</sub> H <sub>10</sub>	C <sub>2</sub> H <sub>4</sub>	(*)	(*)	(*)	<.01	.01
C <sub>5</sub> H <sub>12</sub>	C <sub>3</sub> H <sub>8</sub>	(*)	(*)	(*)	<.02	.03
C <sub>6</sub> H <sub>14</sub>	C <sub>4</sub> H <sub>10</sub>	(*)	(*)	(*)	<.01	.01
COS	COS	(*)	(*)	(*)	<.02	.03
SO <sub>2</sub>	SO <sub>2</sub>	(*)	(*)	(*)	<.01	.01
N <sub>2</sub> (b, difference)	N <sub>2</sub> (b, difference)	95.3	96.4	95.6	88.64	88.04
Flow Rate, SCFH at top of bed	Flow Rate, SCFH at top of bed	500	669	552	643	585
Flow Rate, SCFH (corrected for coal moisture)	Flow Rate, SCFH (corrected for coal moisture)	.665	.728	.265	.677	.258
Moisture in Exit Gas	Moisture in Exit Gas	.008	.010	.008	.008	.008
Tar, lb/hr	Tar, lb/hr	0	.0149	0	.106	.092
Precipitated Coal, lb/hr	Precipitated Coal, lb/hr	(*)	6.96	9.14	9.89	5.31
Percent Precipitation	Percent Precipitation	19.5	18.6	10.0(*)	5.71	5.67
Meanal Solids Retention Time, min	Meanal Solids Retention Time, min	(*)	15	15	14.1	14.0
Duration of Steady-State Period, hr	Duration of Steady-State Period, hr	11	5	3.75	14.1	2.0
Total Product, Pounds	Total Product, Pounds	70	132	34	143	10.4

(\*) Standard conditions are 29.92 in Hg and 70°F.  
 (a) Uncertain because of agglomerate in reactor.  
 (b) Not measured.  
 (c) 9.3% based on original raw coal.  
 (d) In draft tube.

TABLE I

## Properties of Pittsburgh Seam Coal and Products

Run Number	Pittsburgh Seam		IP		2P2		-IP	
	Temperature, °F	Hydrogen, Wt % (dry basis)	Wt %	Density (t)	Wt %	Density	Wt %	Density
Carbon	72.24	70.51	16.3	50.6	16.1	42.4	13.8	47.7
Nitrogen	1.35	1.45	31.0	57.5	27.3	51.5	24.2	51.5
Oxygen	3.55	4.05	31.1	71.2	31.0	56.5	29.2	58.8
Sulfur	11.36	11.12	1.8	76.8	1.5	73.1	7.8	70.2
Volatile Matter	40.1	32.7	0.5	76.8	2.1	74.0	5.5	70.9
Pyritic Sulfur	2.53	1.76						
Sulfate Sulfur	.07	.08						
Size, Tyler Mesh								
+84			0	81.0	3.4	42.0	4.0	41.2
24 x 28	11.2	11.2	16.3	50.6	16.1	42.4	13.8	47.7
28 x 35	35.4	35.4	31.0	57.5	27.3	51.5	24.2	51.5
35 x 48	34.8	34.8	31.1	71.2	31.0	56.5	29.2	58.8
48 x 65	16.9	16.9	1.8	76.8	1.5	73.1	7.8	70.2
65 x 100	1.2	1.2	0.5	76.8	2.1	74.0	5.5	70.9
-100								
Mean Diameter, in (s)			.0165		.0174		.0169	.0156
Mean Density, lb/ft <sup>3</sup> (s)			81.0		81.6		83.1	86.4
Maximum Fluidity DM (s)			> 37,000		16		1.0	

Run Number	Ireland Mine		PR1		PR1A		PR1R	
	Temperature, °F	Hydrogen, Wt % (dry basis)	Wt %	Density	Wt %	Density	Wt %	Density
Carbon	69.88	70.37	675	4.64	750	3.52	775	3.52
Nitrogen	1.25	1.08	70.37	10.2	70.44	10.2	70.60	10.2
Oxygen (by difference)	6.69	6.29	1.24	76.8	4.23	76.8	1.35	76.8
Sulfur	12.66	13.02	6.29	76.8	4.23	76.8	4.92	76.8
Volatile Matter	41.3	35.5	13.02	76.8	15.02	76.8	16.10	76.8
Size, Tyler Mesh								
+28								
28 x 35								
35 x 48								
48 x 65								
65 x 100								
100 x 150								
-150								
Mean Diameter, in (s)			.00774		.00914		.00923	
Mean Particle Density, lb/ft <sup>3</sup> (s)			82		82		82	

(t) Measured in mercury at 1 atmosphere.

(s) Arithmetic mean.

(a) Reciprocal mean.  
(s) As measured in Glassler Plastometer  
(ASTM Method D-2639-67T).

**TABLE 8**  
**Material Balances for Pittsburgh Seam Coal Preoxidation Runs**  
 Basis: 100 lb Dry Coal Fed

Run Number	2PZ	4P	PR1	PR1B
Preoxidized Coal	93.30	97.85	94.05	92.23
Overhead Fines	.58	1.21	.72	1.95
Tar	.20	0	1.01	1.63
Carbon in CO + CO <sub>2</sub>	3.65	2.23	.67	1.17
Hydrogen in H <sub>2</sub> O	1.10	.33	.73	.52
Hydrocarbons (C <sub>1</sub> -C <sub>3</sub> )	.34(1)	.15(1)	.17	.88
Sulfur in SO <sub>2</sub> and COS	.75(2)	.26(2)	.03	.09
Coal Oxygen to Products	0	0	1.70	1.39
Carbon and Sulfur in Condensate	N.D.	N.D.	.10	.10
Total	99.92	102.03	99.18	99.96

(1) Only CH<sub>4</sub> was determined.  
 (2) By sulfur balance on coal feed and preoxidized coal product.

TABLE 9

Distribution of Oxygen in Products of Preoxidation of Pittsburgh Seam Coal

Run Number	2P2		4P		PR7		PR1B	
	Lb/hr	Percent	Lb/hr	Percent	Lb/hr	Percent	Lb/hr	Percent
<b>Output</b>								
To Water	.648	46.6	.235	25.1	.602	76.7	.229	57.5
Condensate	.009	.6	.006	.6	.008	1.0	.006	1.5
Moisture in Gas	.546	39.3	.437	46.7	.133	16.9	.118	29.5
To CO <sub>2</sub>	.080	6.5	.058	6.2	.026	3.4	.029	7.3
To SO <sub>2</sub>	.056	4.0	.024	2.5	0	0	.001	.3
To COS	(1)	-	(1)	-	.002	.2	.002	.5
In Tar	0.0	-	0.0	-	.002	.2	.013	3.4
Unreacted O <sub>2</sub>	.040	3.0	.178	18.9	.012	1.6	.0	.0
On Coal (by diff.)					0.0	0.0	0.0	0.0
Total	1.389	100.0	.937	100.0	.785	100.0	.398	100.0
<b>Input</b>								
From Coal (by diff.)	0		0		.177		.078	
Lb/hr O <sub>2</sub> from Air	1.389		.937		.608		.320	
Total	1.389		.937		.785		.398	

(1) Not determined.

TABLE 10

Effect of Pretreatment Pressure on  
Decaking of 28 x 200 M Ireland Mine Coal

<u>Pretreatment Pressure, atm</u>	<u>Run No.</u>	<u>Pretreatment Temperature, °F</u>	<u>Wt. % Preoxidation</u>	<u>Pretreated Coal<sup>(1)</sup> Max. Fluidity, DDPM</u>
1.5	1A	700	5.5	9
1.5	2A	700	13.8	0
15	1P	700	19.5	16
15	2P2	750	18.6	1.0

(1) Via Gieseler plastometer (ASTM Method D-2639-67T).

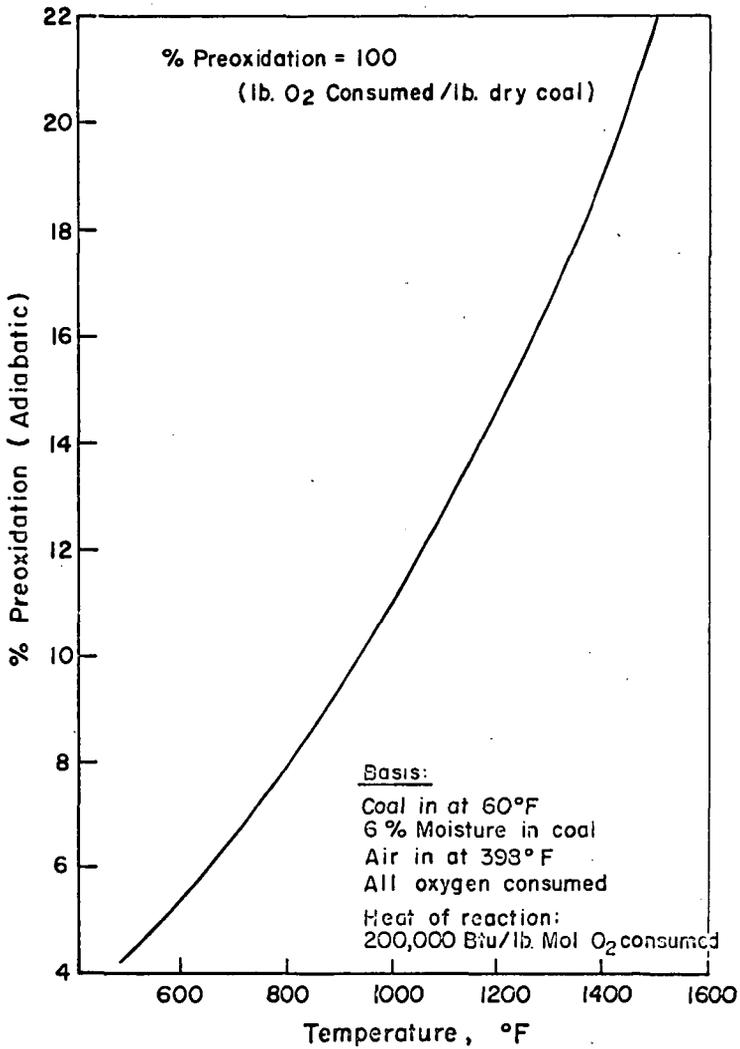


Figure 1 - % Preoxidation vs Temperature  
For Adiabatic Constraint.

FIGURE 2  
FLOW DIAGRAM FOR COAL PRETREATMENT

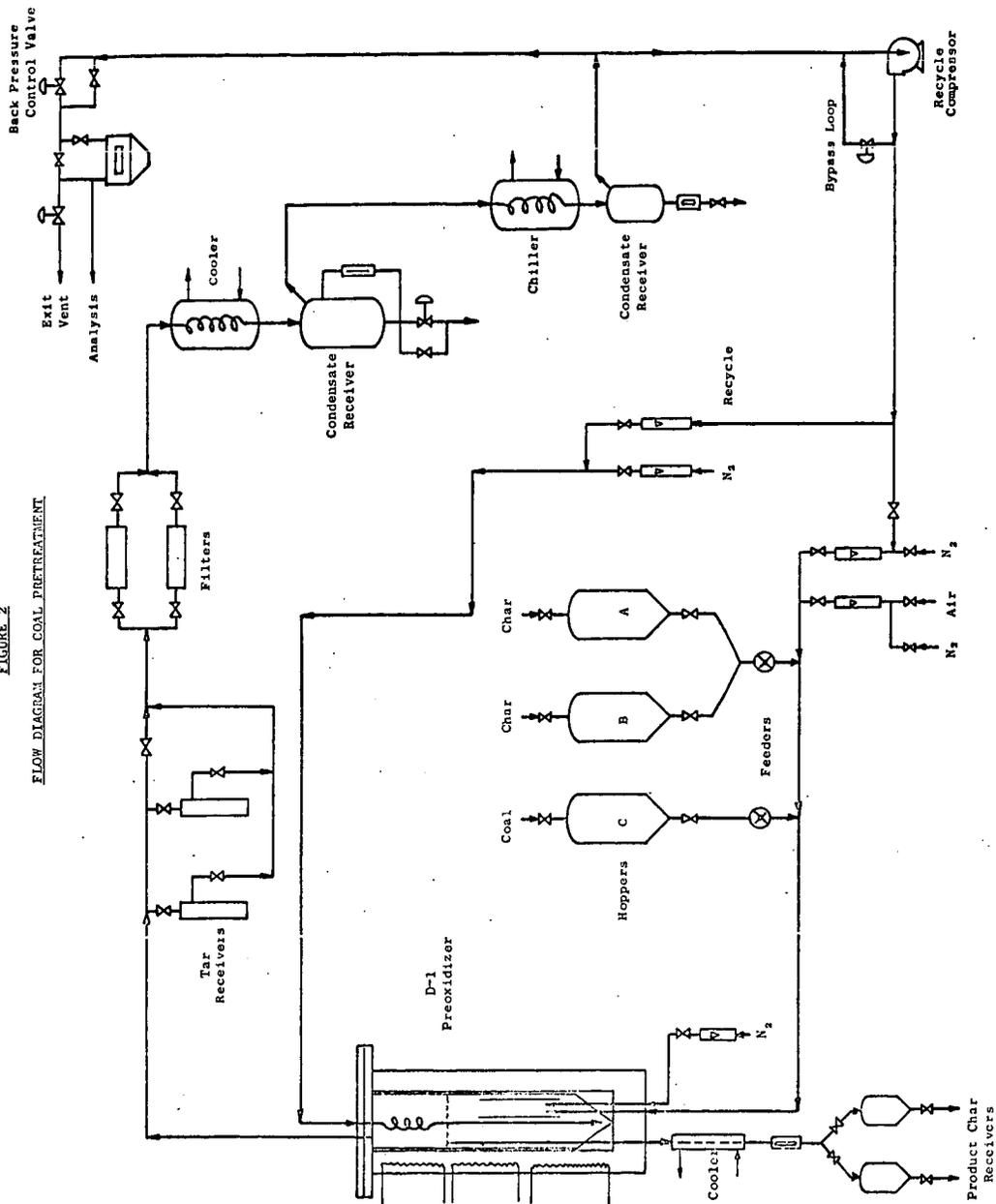


FIGURE 3  
CONFIGURATION OF DRAFT TUBES USED IN SEEDED COAL TESTS

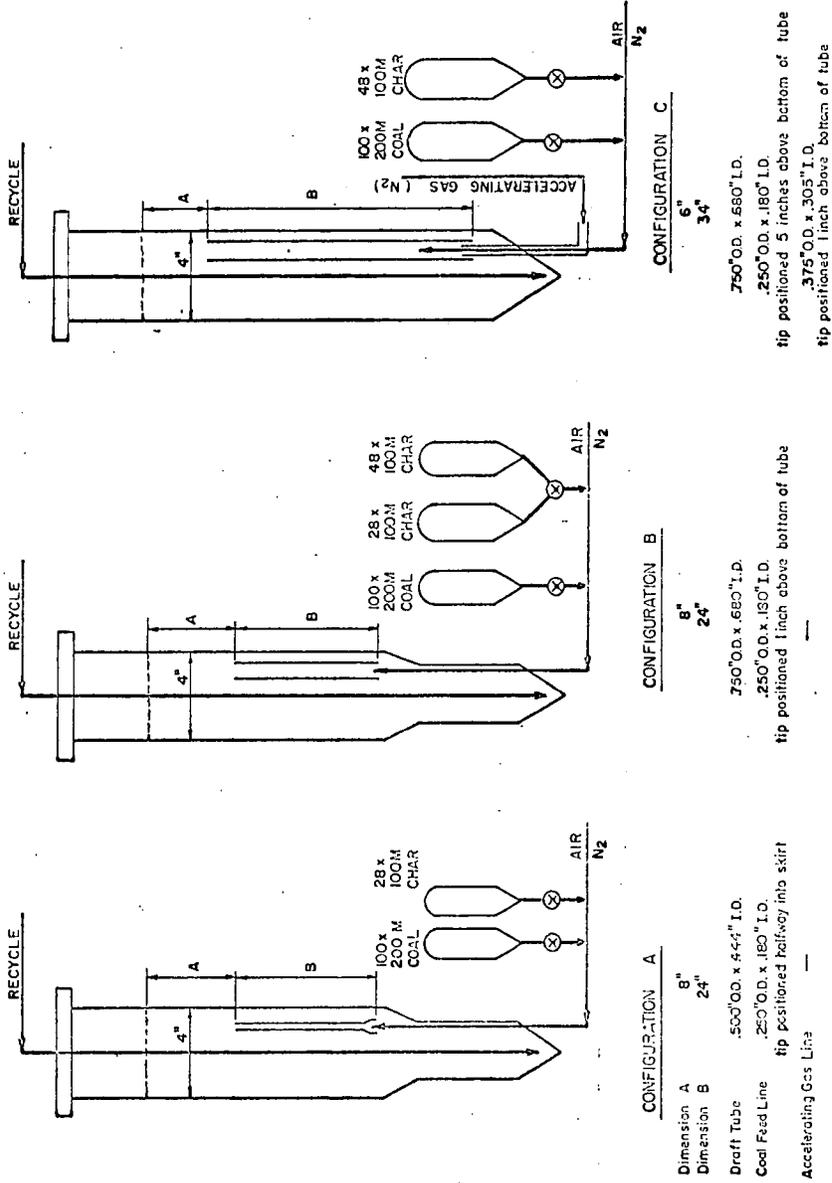
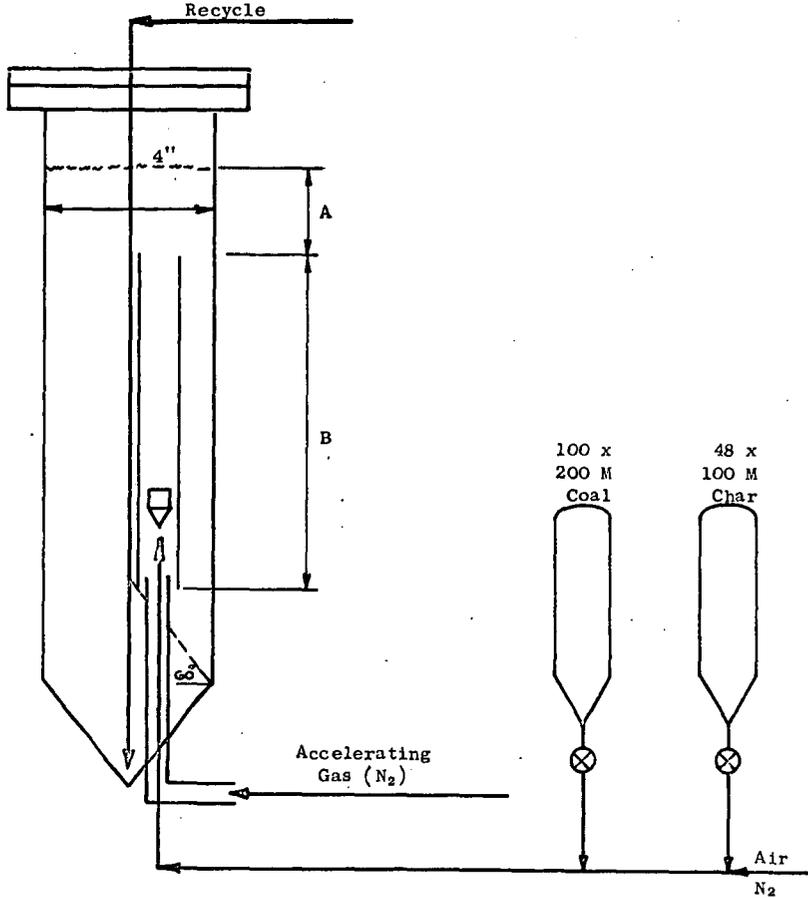


FIGURE 4

Configuration of Draft Tube Used in Seeded Coal TestsCONFIGURATION D

Dimension A	4"
Dimension B	34"
Draft Tube	.750" O.D. x .630" I.D.
Coal Feed Line	.250" O.D. x .180" I.D.
	Tip positioned 5-inches above bottom of tube
Accelerating Gas Line	.375" O.D. x .305" I.D.
	Tip positioned 1-inch above bottom of tube
External Baffle	3-5/8" x 1-3/4"
	Elliptical, 60° from the horizontal
Internal Baffle	3/8" D x 60° Cone
	Tip positioned 1/2" above coal feed line

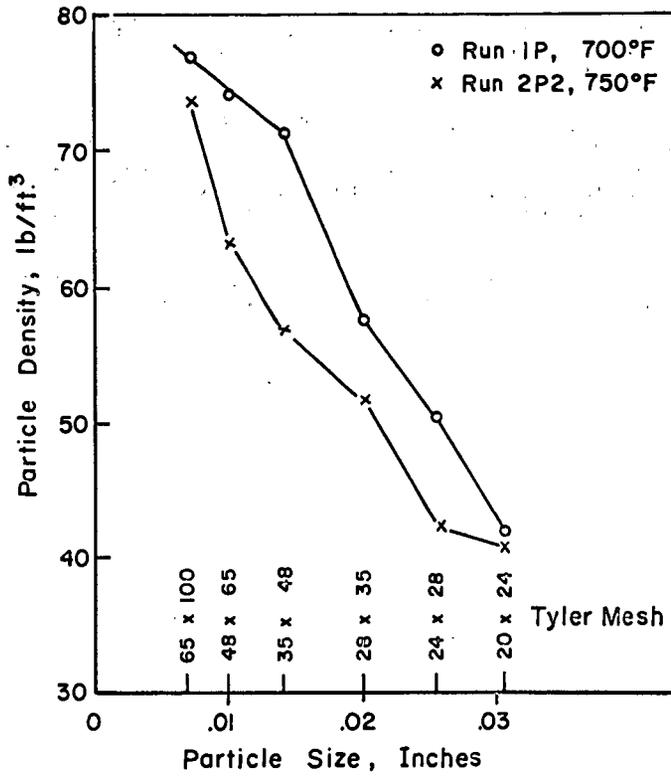


Figure 5 - Particle Densities For Preoxidized Ireland Mine Coal.