

FORM-COKE REACTIVITY AND IT'S EFFECT ON BLAST FURNACE OPERATION

Robert T. Joseph

FMC Corporation
Chemical and Development Center
Princeton, New Jersey 08540

A study was carried out to compare the effect of carbon reactivity on the operation of commercial iron blast furnaces. Two cokes, produced via by-product oven technology with a relative reactivity in carbon dioxide of one where contrasted with a carbon reductant produced as a form-coke briquet from an Intermountain, noncoking coal with a relative reactivity of twenty. The work was carried out both in a British and an American production blast furnace operating under commercial conditions across a protracted period. The information obtained by reflectance analysis offers an explanation of what appears to be an advantage for high reactivity form-cokes.

CLEAN COKE PROCESS: FLUID-BED
CARBONIZATION OF ILLINOIS COAL

T. F. Johnson, K. C. Krupinski, and R. J. Osterholm

U. S. Steel Corporation
Research Laboratory
Monroeville, Pa. 15146

The CLEAN COKE Process is based on a conceptual plan, developed by United States Steel Corporation, for converting nonmetallurgical-grade coals to metallurgical coke, chemical feedstocks, and liquid and gaseous fuels (1). In accordance with the process, low-temperature carbonization of coal and coal hydrogenation are combined in a manner that affords an energy- and hydrogen-balanced process. Moreover, coke production—from initial carbonization to finished product—is implemented so as to avoid contamination of the atmosphere with pollutants.

The process is shown schematically in Figure 1. Mined coal is beneficiated, and a portion is sent to a low-temperature carbonization plant where pyrolysis occurs at pressures as high as 165 psia in a fluidized-bed system. A stream of cleaned, hydrogen-rich recycle gas serves as the fluidizing medium in the carbonizer vessel. The high partial pressure of hydrogen in the gas and the essential absence of sulfur impurities provide an effective hydrodesulfurization environment whereby the sulfur content of the coal is reduced from an initial level of more than 2 percent to below 0.5 percent in the resulting char. The char can then be pelletized with a process-derived binder oil, and the pellets can be cured and calcined to produce the low-sulfur metallurgical coke.

The second portion of the beneficiated coal is sent to a hydrogenation plant where the coal is slurried with a process-derived carrier oil and subjected to noncatalytic hydrogenation at elevated temperature and pressure. Liquid product is separated from unreacted coal and mineral matter in a vapor-stripping vessel where the hydrogenate is contacted countercurrently with a stream of hot process gases and the volatile matter removed. Residual solids pass through a lock system into a receiving vessel and are quenched and withdrawn from the system.

In the coke-preparation section, carbonization char and liquid binder, the latter obtained from liquids treatment, are combined in a disc pelletizer to form green pellets. These are then cured by heating in the presence of air to impart a degree of green strength, and the cured pellets are finally calcined at temperatures up to 2000 F (1100 C) to produce the hard metallurgical coke pellets.

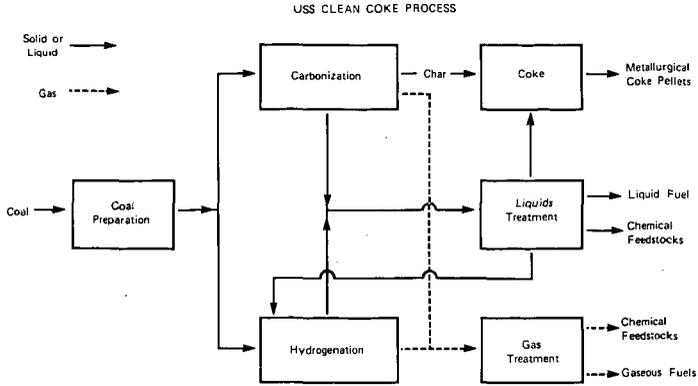


Figure 1

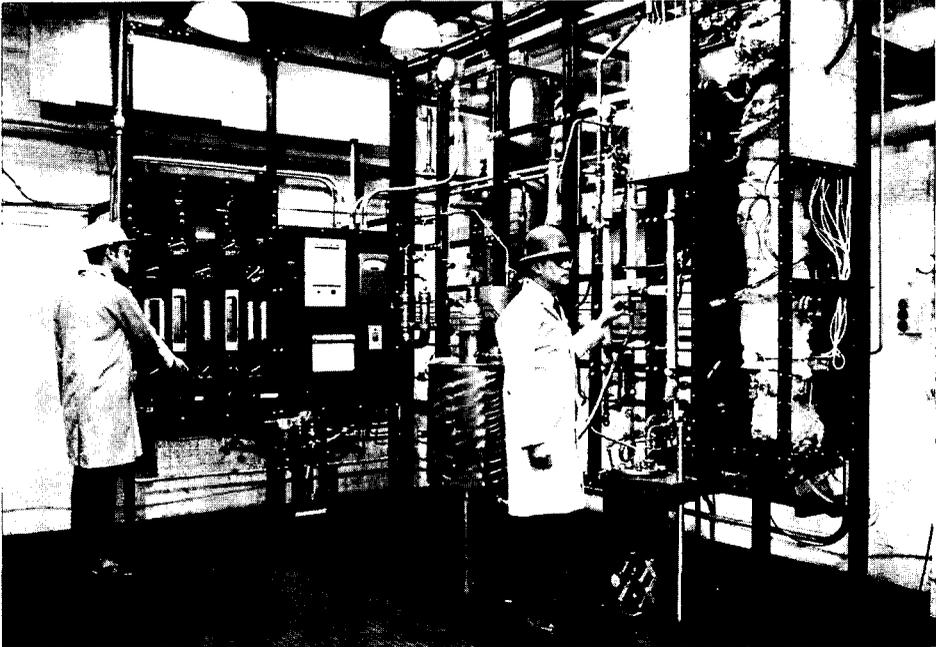


Figure 2 Three-inch bench-scale carbonizer.

This paper summarizes the results of studies on carbonization of Illinois No. 6 seam coal in a fluidized bed to produce the low-sulfur char that is ultimately converted to metallurgical coke. The objectives of the carbonization studies were to determine the efficiency of desulfurization of coal and to determine the effect of carbonization variables—temperature, pressure, reaction time, and feed pretreatment—on the desulfurization reaction (2).

Bench-Scale Carbonization Reactor

For this work, a special pressurized reactor was designed and fabricated to permit carbonization-desulfurization of coal in a closed loop with recycle of gas under pressure. The bench-scale unit, shown in Figure 2, has a temperature limit of 1400 F (760 C) and a pressure limit of 140 psia. The reactor section is constructed of 3-inch, Schedule 40, Type 316 stainless-steel pipe in the fluid-bed region and expands to 6-inch pipe above the fluid bed. A perforated stainless-steel plate serves both as a gas-distributor plate and as the bed support. The fluidizing gas is heated by an electric heater and the external heater windings on the reactor section. The hot gas leaving the reactor is cleaned of any coal or char particles by the Pyrex-wool filter. After removal of the solids, heavy oils are condensed from the gas in a water-cooled tar trap, and lighter oils are condensed in a dry-ice trap. Hydrogen sulfide is removed by the H₂S absorber, which consists of a 2-inch stainless-steel U tube packed with Molecular Sieves. Gas flow is monitored by flow meters, and the gas is recycled by an air-driven Haskel AGD-4-C double-acting, single-stage gas booster pump. As additional gas is generated during a carbonization, a constant pressure is maintained by means of a gas accumulator. Water is released from the gas accumulator, thereby increasing the volume of the system; no gas is vented. Recycled gas is analyzed for hydrogen, methane, and carbon monoxide by a Greenbrier process gas chromatograph, which is permanently connected to the carbonizer.

Experimental Procedure

A typical test consisted of charging the reactor with 1 kg of coal or a blend of 400 g of recycle char and 400 g of coal; this gave an expanded bed height, during fluidization, of about 18 inches. A synthetic mixture of hydrogen, methane, and carbon monoxide (approximately the steady-state recycle-gas composition) was added to the reactor to the desired test pressure, and the gas flow rate was adjusted to give a superficial gas velocity of about 1.2 feet per second in the reactor. The recycle gas and the reactor were then heated to the desired temperature, and the fluid bed was maintained at that temperature for the specified residence time. After the reactor cooled, the reaction products were removed and analyzed.

Product water was separated from the carbonization tar by centrifugation to allow determination of the types and amounts of products available from the carbonization tar. The tar was distilled at a pressure of 200 torr by using a Vigreux column 2.5 cm in diameter

by 25 cm long. Three fractions were obtained: (1) a chemical oil, distilling to 385 F (195 C); (2) a middle oil, distilling at 385 to 635 F (195 to 335 C); and (3) a heavy oil (residue), distilling above 635 F. The chemical oil was further resolved into a tar-acid fraction, a tar-base fraction, and a neutral-oil fraction by extraction with aqueous caustic and acid.

The coal and char were analyzed by standard ASTM methods. The tars and derived oils were characterized by gas chromatography (GC); tar acids and bases were also determined by a potentiometric titration procedure. Mass spectroscopy and GC were used to analyze the gaseous products.

Results of Typical Carbonization of Illinois Coal

A typical carbonization of the coal feedstock resulted in removal of 80 percent of the contained sulfur and gave the following yield of products, in weight percent of the charge: char, 66.4; tar, 13.9; gas, 14.6; and water, 5.1. These results were obtained with a preoxidized feed coal and with the carbonizer operated at 1365 F (740 C) and a pressure of 80 psia for a 30-minute residence time. Efficiency of sulfur removal is illustrated by the reduction of sulfur content in the solid materials from 1.7 percent in the coal to 0.5 percent in the char product.

Figure 3 illustrates stream flow and product distribution in a typical carbonization test. Experimentally, the yields of char and gas are readily measured, but yield of liquids is difficult to measure quantitatively because of mechanical problems in removing water and tar from the unit. Consequently, handling losses were distributed proportionately between water and tar to provide overall material balance.

Results from analysis of the char product are presented in Table I, along with corresponding results for the feed coal. The sulfur content of the char (0.5%) is significant, since this indicates that the char can be converted to a coke product containing less than 0.5 percent sulfur. Of the sulfur removed from the coal, about 85 percent was converted to hydrogen sulfide, and the rest was separated as volatile organic sulfur compounds found mainly in the tar.

The gas product, after removal of hydrogen sulfide, had three major constituents: hydrogen, methane, and carbon monoxide in the molar ratio of 40:52:8. Small amounts of ethylene, ethane, propane, and carbon dioxide were also identified, although none was present in significant concentration.

To determine its chemical composition, the carbonization tar was distilled to divide it into 20 percent chemical oil, 37 percent middle oil, and 43 percent heavy oil. The chemical oil was then extracted to separate tar acids, tar bases, and neutral oil, and individual components were identified by gas chromatography. By

Table I

Analyses of Feed Coal and Product Char

<u>Analysis, wt %</u>	<u>Coal</u>	<u>Char</u>
Proximate		
Volatile matter	34.3	1.60
Fixed carbon	59.5	89.18
Ash	6.2	9.22
Ultimate		
Carbon	75.48	86.58
Hydrogen	5.21	1.64
Nitrogen	1.72	1.27
Oxygen	9.71	1.52
Sulfur	1.74	0.50
Ash	6.14	8.49

type of compound, the chemical oil consists of about 55 percent phenols, 20 percent benzenes, 7 percent each of tar bases and naphthalene, with the balance being naphthenes and heterocyclics.

The water produced during the carbonization reaction contains compounds typically found in waste water from conventional coke-oven operations. The component analysis of the water is given in Table II and shows a dissolved ammonia content of about 3 percent and a tar-acid content of almost 0.6 percent. Dissolved salts, including the thiocyanates, cyanides, sulfates, and sulfides, total about 3800 ppm. Cleanup of this stream will be necessary, but it will be simplified in this process by confining all the discharged water to a single stream.

Effects of Carbonization Reaction Conditions

A study was made to assess the effects of residence time, temperature, and pressure on the carbonization reaction and products. This study was limited by the relatively slow heat-up rate of the 3-inch unit, in which about 70 minutes was required to reach 1150 F (620 C) and about 100 minutes to reach 1365 F. For convenience in measuring times, heat-up periods were excluded, and residence times were measured only from that point at which the fluid bed reached the desired test temperature. However, it was recognized that intermediate temperatures above about 800 F (425 C) contributed an unmeasured, but significant, effect to residence time. To study the effect of heat-up time on coal desulfurization, a test was performed at nominal zero residence time, 1365 F, and 80 psia. The results from this test,

Table II

Analysis of Product Water From Carbonization

<u>Component</u>	<u>Parts per Million</u>
SCN ⁻	1,171
CN ⁻	74
SO ₄ ⁼	2,050
S ⁼	562
NH ₃	33,320
Phenol	3,200
o-Cresol	700
m,p-Cresol	1,500
Xylenols	500

along with results obtained with the 30-minute residence time, are shown in Table III. Gas compositions and char yields were very similar for the two tests; the hydrogen content of the gas was about 38 percent, and the char yield was 66 percent. The sulfur content of the char at 0- and 30-minute residence times was 0.73 and 0.51 percent, respectively. The data indicate that the coal is devolatilized and significantly desulfurized during the heat-up period. However, increased desulfurization of the char is achieved by the additional residence time at the reaction temperature.

Table III

Effects of Time and Temperature on Desulfurization of Coal at 80 psia

Temperature, F	1365	1365	1150
Time at Temp, min	0	30	30
Fluidizing-Gas Composition, mole %			
H ₂	38	40	27
CH ₄	52	52	} 73
CO	10	8	
Sulfur Content, wt %			
Coal	1.76	1.76	1.76
Char	0.73	0.51	0.85

The effect of temperature on coal desulfurization was determined by carbonizing the preoxidized coal at 1150 F and 1365 F (80 psia and 30 minutes). The results of these tests, also in Table III, show the improvement in desulfurization and the hydrogen enrichment of the recycle gas with increasing temperature. However, in considering the slow rate of heat-up, it is seen that about 70 minutes of heating time is required to reach 1150 F, and 100 minutes is required for 1365 F. Therefore, a comparison of an 1150 F, 30-minute carbonization with a 1365 F, 0-minute carbonization may be more valid, since the total heat-up time plus residence time is the same. The sulfur content of the char produced at 1365 F and 0 minutes is 0.73 percent, which is lower than the 0.85 percent sulfur for the 1150 F, 30-minute char. This demonstrates that an increase in temperature does indeed improve desulfurization of the coal.

A series of three carbonizations of preoxidized coal was conducted at 1365 F and pressures of 30, 80, and 125 psia to study the effect of pressure on the desulfurization of the coal. Nominal residence time for these tests was 30 minutes. Desulfurization of the coal improved with increasing operating pressure, and steady-state gas data showed, as expected, decreasing hydrogen and increasing methane contents with increased operating pressure. Summary data for these carbonization tests at 30, 80, and 125 psia are presented in Table IV and demonstrate a definite advantage for higher-pressure operation with respect to desulfurization of the coal. Corresponding char products contained 0.77, 0.50, and 0.33 percent sulfur. The improvement in sulfur removal is the result of higher partial pressure of hydrogen, which of course increased in this series although the hydrogen concentration in the recycle gas decreased with higher pressures.

Table IV
Effect of Pressure on
Desulfurization of Coal at 1365 F

Pressure, psia	Recycle-Gas Composition, mole %			Sulfur Content, wt %	
	H ₂	CH ₄	CO	Dry Coal	Char
30	54.4	34.4	10.9	1.74	0.77
80	39.4	51.8	8.5	1.74	0.50
125	32.4	60.0	7.2	1.74	0.33

Studies on Modification of Feed Coal

Modification of the coal feed for fluid-bed carbonization tests was done to ensure that the fluid nature of the bed was not lost during the heat-up period. A fluid bed is essentially a suspension

of solid particles (coal or char or both) in a flowing gas stream. Because all the particles are in motion, a fluid bed exhibits many of the properties of a boiling liquid, including rapid heat transfer throughout the mass (3). Also, the fluid nature permits continuous addition of feed and withdrawal of product, which facilitates continuous operation. However, when a caking coal, such as Illinois No. 6 seam coal, is heated to about 750 F, the solid particles of coal become plastic and stick together in an agglomerated mass that destroys the fluid state.

Agglomeration of caking coals can be avoided by mild pre-oxidation to chemically alter the surface of the coal particles, or by blending the coal with a noncaking char, which physically separates the softened coal particles in the plastic region (4,5).

The effect of coal preoxidation on desulfurization and tar composition was determined by carbonizing unoxidized and preoxidized coal at 1365 F and 80 psia. Unoxidized coal was carbonized with an equal amount of recycle char to avoid agglomeration. For control purposes, a sample of the recycle char was carbonized neat to establish the extent of desulfurization and gasification that occurs under conditions of treatment. The results of the control test, given in Table V, show a reduction in sulfur content from 0.88 to 0.49 percent and a weight loss of 1.2 percent. The results of the test with a blend of unoxidized coal and recycle char are also presented in Table V, and show an apparent 88 percent desulfurization. On the assumption of no interactions between recycle char and feed coal, it is calculated that char produced from the coal portion of the carbonizer feedstock contained only 0.34 percent residual sulfur. Whether or not interactions between char and coal were actually occurring during carbonization, the char sulfur content was still at a low level and certainly did not exceed the 0.43 percent value determined for the total char.

A comparison of the carbonizations of unoxidized and pre-oxidized coal at 1365 F, 80 psia, and 30 minutes residence time is also given in Table V. Carbonization of the unoxidized coal gave apparently better desulfurization, less char, and more tar than carbonization of preoxidized coal; however, these results represent a single set of data and may only indicate the effect of small variations in reaction time and temperature.

Feed coal for these tests was preoxidized in the 3-inch carbonizer by using heated air as the fluidizing medium. Treating conditions included atmospheric pressure, a temperature of 350 F (175 C), and a residence time of about 10 minutes. When coal/char blends were used to prevent bed agglomeration, char from previous carbonizations were used in a weight ratio of 1 to 1.

Table V

Effects of Feed Modification
on Desulfurization of Coal

	<u>Preoxidized Coal</u>	<u>Unoxidized Coal + Char</u>	<u>Char</u>
Yield Data, wt %			
Char	66.4	61.3	98.8
Tar	12.2	15.5	-
Water	3.3	5.0	-
Gas + loss	18.2	18.2	1.2
Sulfur Analysis, wt %			
Coal	1.76	1.79	-
Starting char	-	0.88	0.88
Final char	0.51	0.43	0.49

Continuous Carbonization of Coal in Two Stages

Both preoxidation of coal and blending of coal with recycle char effectively eliminated bed agglomeration in batch tests, which were limited by a rather slow heating rate of 10 degrees per minute. However, subsequent tests on continuous feeding of preoxidized coal to a hot fluid bed resulted in agglomeration of the bed, because continuous feed addition results in a much faster heating of the added coal. The heating rate in continuous operation appears to be about 10 degrees per second, which generates sticky tars at a rate faster than that at which tars can be vaporized away from the bed, and agglomeration results.

Subsequent tests on continuous feeding led to the development of a two-stage system, involving partial devolatilization of oxidized coal in a fluid bed operating at temperatures in the range 800 to 840 F (425 to 450 C), followed by carbonization in a fluid bed at temperatures of 1300 to 1400 F (700 to 760 C). A sample of 20- by 65-mesh Illinois coal was stage-carbonized in the 3-inch bench unit. In both devolatilization and carbonization stages, the coal was added to the top of the bed, using a rotary feed valve. Recycle gas was the fluidizing gas in both stages. During the first stage, 200 g of coal that had been oxidized at 350 F was added at the rate of 14 g per minute to a fluid bed of 800 g of coal which had been held at 840 F for 10 minutes. After all the coal was added, the bed was maintained at temperature for 20 minutes at 125 psia. The devolatilized coal (semichar) removed weighed 760 g and contained 16 percent volatile matter, an indication that about 218 g of volatiles had been removed from the coal; handling loss was about 18 g.

A portion (520 g) of the semichar was returned to the reactor and heated to 1380 F (750 C) at 125 psia. After a 15-minute hold, 200 g of the semichar was added to the fluid bed at 14 g per minute. When all the coal was added, the bed was maintained at temperature for 15 minutes.

The recovered char weighed 557 g and contained 1.86 percent volatile matter, an indication that about 104 g of volatiles was removed from the partially devolatilized coal; solids loss was about 50 g, which includes some handling loss and partial gasification of the fixed carbon.

Figure 4 is a summary of this staged carbonization and includes the yield of product at each stage, corrected for losses. The figure indicates that the yield of semichar was 78.2 percent of the preoxidized feed in the first stage, and about 63 percent of the volatile matter was removed. The product char from the second stage was obtained in 66.9 percent yield overall.

Results of proximate and ultimate analyses for the oxidized coal and the products of each stage of carbonization are presented in Table VI. Desulfurization was significant during the first stage, a decrease in sulfur content from 2.28 percent to 1.63 percent. The sulfur content of the char was 0.33 percent. The nitrogen content varied from 1.67 to 2.05 to 1.49 percent, an indication that nitrogen compounds react in the final carbonization stage almost exclusively.

Table VI
Analysis of Products of
Staged Carbonization of Coal

	<u>Preoxidized</u>	<u>Partially Devola- tilized</u>	<u>Carbonized</u>
Temperature, F	350	840	1380
Pressure, psia	15	125	125
Analysis of Products, wt %			
Proximate			
Volatile Matter	34.31	15.96	1.86
Fixed Carbon	58.72	74.50	87.43
Ash	6.97	9.54	10.71
Ultimate			
Sulfur	2.28	1.63	0.33
Carbon	72.96	74.80	86.29
Hydrogen	5.07	3.97	1.66
Nitrogen	1.67	2.05	1.49

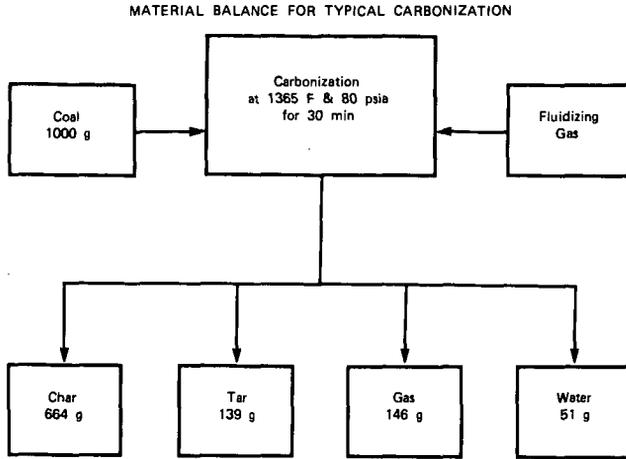


Figure 3

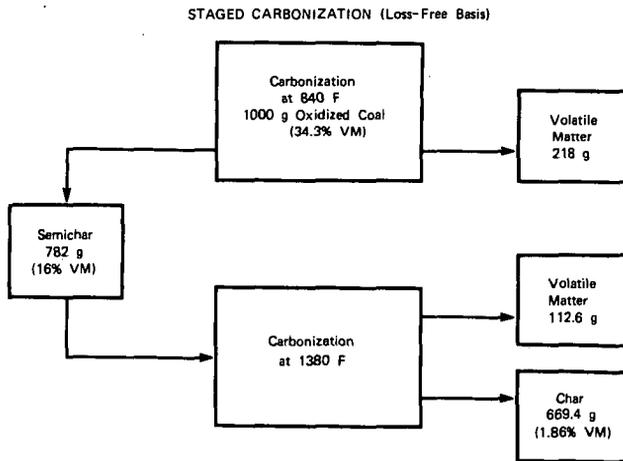


Figure 4

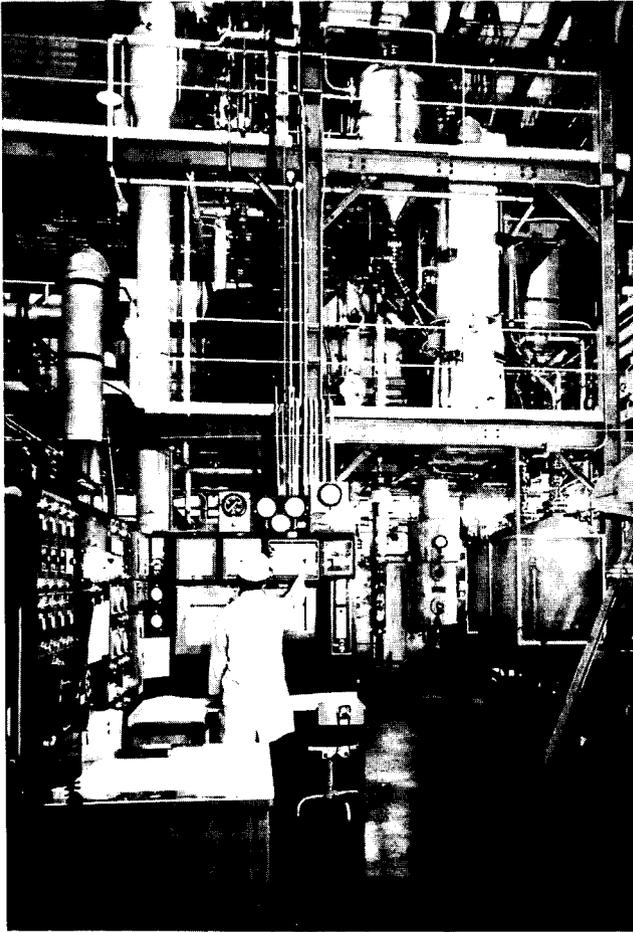


Figure 5 Carbonization process-development unit
for CLEAN COKE project.

Current work on the CLEAN COKE Process is being conducted in a 500-pound-per-day process-development unit (PDU). The carbonization PDU, Figure 5, consists of a 10-inch-diameter fluid-bed reactor with lock hoppers for coal feed and char product, and gas-cooling and -scrubbing equipment for recovery of heat and tar from the recycle gas. Preliminary testing and shakedown runs in the PDU have been completed, and carbonization tests are now in progress to provide data on continuous operation in the unit. Results from these scale-up tests will be published when the work is complete.

The work described here is supported in part by the Energy Research and Development Administration, under contract No. 14-32-0001-1220.

References

1. K. A. Schowalter and N. S. Boodman, "The Clean Coke Process for Metallurgical Coke," Chemical Engineering Progress, Vol. 70, No. 6, June 1974.
2. USS Engineers and Consultants, Inc., "Clean Coke Process - Summary of Bench-Scale Studies," Interim report for period March 1972 - April 1974, Contract No. 14-32-0001-1220, Prepared for U. S. Department of Interior, Office of Coal Research, August 1974.
3. F. A. Zenz and D. A. Othmer, "Fluidization and Fluid-Particle Systems," Charles R. Wilke, Consulting Editor, Reinhold Publishing Corporation, New York, 1960.
4. P. J. Wilson and J. H. Wells, "Coal, Coke, and Coal Chemicals," McGraw-Hill, New York, 1950.
5. FMC Corporation, "Char Oil Energy Development," Final report for period June 1962 - December 1965, Contract No. 14-01-0001-235, Prepared for U. S. Department of Interior, Office of Coal Research, December 1965.

FORMCOKE PREPARATION IN
CLEAN-COKE PROCESS

R. W. Shoenberger

Senior Research Engineer-Raw Materials Utilization Division

Research Laboratory
U. S. Steel Corporation
Monroeville, Pennsylvania 15146

For presentation at the Symposium on New Coke Processes
Minimizing Pollution, Division of Fuel Chemistry, ACS,
Chicago, Illinois, August 1975, and for publication as
preprint for distribution in advance to Division members.

The Clean-Coke Process being developed by U. S. Steel under contract with the U. S. Government (Energy Research and Development Administration) has been described previously.^{1)*} Chemicals, fuels, and coke are the major products. However, an important phase of the Clean-Coke project is the ability to make an acceptable metallurgical coke from desulfurized char and heavy residual oils from the process. Of the various types of agglomeration (briquetting, pelletizing, nodulizing, or extrusion) considered, pelletizing appeared to have the best potential for more easily producing a superior product at the most reasonable cost by using char and binder derived from the process. The char as characterized in bench scale studies is low in density with a large percentage of cenospheres. Binder is available in large quantities. An advantage of producing coke by such a formcoke process is the reduction of pollution problems normally encountered in the conventional coke making process. The steps required for coke preparation will be carried out in a closed system with the off gases collected and returned to be processed through a common system. As a result, atmospheric pollution is practically non-existent.

Because the fluid-bed char-making unit was not yet constructed to produce desulfurized char at the time the coke-making phase of the process was initiated, char was produced in a

* See References.

four-inch fluid-bed carbonizer using nitrogen as the fluidizing gas. Also, simulated binder was used because a binder was not available from the process in the quantity required for the coke-making studies. This paper presents the development of this method and shows some of the factors influencing the resultant coke properties.

Materials

Illinois No. 6 Seam coal, obtained from Franklin County, Illinois and washed at a 1.35 specific gravity, was charred in a fluid bed to a volatile matter content of 9 to 12 percent. This char was made by batch operation at a final bed temperature of 1150 F in an air-nitrogen fluidizing gas. Photomicrographs of the char are shown in Figure 1. The coal and char were nominally minus 10-mesh in size.

Various binders were used to determine how their properties would affect the resultant coke properties. The characteristics of these binders are listed in Table I. Some of the binders represent the type of binders available from the process whereas others represent binders from other sources having a wide range of properties.

Pelletization

A 24-inch pelletizing disc with a continuous-heated, (300 - 350 F) solids feeding system was used to study the pelletization of char fines with a heavy residual oil binder. The binder

was heated in a surge tank and sprayed on the solids to form the pellets. Figure 2 shows the equipment installed for the present work.

Strong green pellets of the desired size (2- by 3/4-inch) were produced from all test binders by regulating the disc conditions, the temperature of the binder and the solids, and by changing the properties of the binder. Test conditions for typical runs are listed in Table II. The percentages of binder required increased as the fineness of the solid material was increased and generally made up about 30 percent of the mix except for the pellets made with fluxed coal extract. The reason for the need to use 38 percent of the fluxed-coal extract binder with the minus 28-mesh char is not known. The recycle material was reduced to about 10 percent in the later runs. The pellets are soft as they are discharged hot from the disc, but they can fall 3 to 6 feet without changing their shape. When they cool they become hard and have a nominal 350-pound crushing strength. A test in the ASTM shatter machine showed no degradation of cold green pellets at room temperature when dropped four times.

Studies were conducted with coke fines and coal tar pitch to determine factors affecting pelletizing rate. It was necessary to provide multiple sprays and solid feeders for feeding the disc. In short runs, 3.5 pounds per minute of plus 3/4-inch green pellets were produced. Test data indicate that about 12 to 15 tons per hour could be produced on a 20-foot disc.

Pretreatment and Carbonization of Pellets

With no pretreatment (curing) the green pellets agglomerated together during carbonization because the binder became soft and melted. In addition, any shearing action disintegrated the pellets to fines during the heating period while the pellets were soft. Preliminary studies showed that the pellets became soft at about 250 F and then hardened at about 900 F in the absence of oxygen. Tests also showed that pellets would harden permanently at 500 F in the presence of oxygen. Larger scale tests were then conducted in different types of equipment (such as a vertical retort, small test coke oven, and a stationary grate) to simulate the various methods that might be used for treating (curing) the pellets.

Pretreatment of the green pellets had to be initially conducted in shallow beds (two pellet depth) for long treating periods (3 to 5 hours). During these studies, it was found that mixing the pellets with an inert supporting medium reduced agglomeration, permitted deeper beds, and allowed faster heating rates to be used without cracking or destroying the pellets. Char fines or sized coke pellets (up to 3/4-inch top size) were used as media. The following methods were found effective in pretreating the green pellets:

- 1) Indirect heating in a Brennstoff Technik²⁾ type coke oven which is relatively narrow (from 60 to 125 mm wide) and has walls made of iron, resulting in very fast heating rates. Pellets were heated from 900 to 1100 F in 1 and 3/4 hours. Agglomeration was prevented by mixing about 50 percent of 1/8 x 0 char as a supporting medium.
- 2) Direct heating in an intermittent vertical retort with 50 percent of a 1/2- by 1/4-inch supporting coke medium to 900 F in an inert atmosphere and to 500 F in an oxygen atmosphere for 1 and 3/4 hours (tested in bed depths up to 8-feet).
- 3) Direct heating on a grate with 50 percent of a 1/2- by 1/4-inch supporting coke medium to 900 F in an inert atmosphere and to 500 F in an oxygen atmosphere for 1-1/2 hours (tested in bed depths up to 18-inches).

Because shearing of the pellets is minimized in curing on a traveling grate, a grate using a 1/2- by 1/4-inch supporting coke medium and an oxygen atmosphere was selected as the most suitable equipment to use. A curing pot was installed for the present studies. Figure 3 is a diagram of the curing pot system.

When the pellets are cured or pretreated properly, they are sufficiently strong to withstand handling and will not soften upon further heating in the coking step. These treated pellets

have been successfully coked in a continuous coking kiln by using inert gases to coke the pellets. The coked pellets have a good appearance (very few cracks and a metallic, smooth surface) with 97 to 100 percent of the pellets being recovered after coking to 1800 F. The normal yield obtained after driving off the volatiles of the green pellets in both the pretreatment and coking steps is about 70 percent. A method was also found to coke green pellets to 1800 F by using 50 percent 1/8-inch by 0 supporting char medium in a Brennstoff-Technik type coke oven for 5-1/2 hours. However, such a method would be expensive and it would be difficult to control pollution when discharging and quenching the coke and medium. Therefore, a continuous vertical coking kiln was built for the present studies. This kiln is shown in Figure 4.

Properties of Coke Pellets

The tests used to evaluate the quality of the coke pellets were tumbler strength, crushing strength (total force on whole pellet), compressive strength, and apparent specific gravity. Because of the large quantity of sample required for the ASTM tumbler test, the number of tests was minimized. However a good relationship was found between crushing strength and the hardness value (plus 1/4-in.) determined from the tumbler test, Figure 5. A discussion follows on some of the factors affecting the resultant coke properties.

Type of Binder

Table III shows some physical properties of formcoke produced from the various types of binders at similar conditions. The type of binder did have a significant effect on the properties of the resultant formcoke. It was possible to improve the effectiveness of some binders by using additives such as carbon black or coal or by air blowing. The strength of the formcoke generally increased as the carbon to hydrogen ratio and the Quinoline Insoluble content increased in the binder.

Size Consist of Char

The effect of grinding the char to finer sizes is shown in Table IV. As noted, the strength of the formcoke increased as the char was pulverized more finely. When the char containing large cenospheres and an open structure (see Fig. 1) is used in preparing the pellets, a weak formcoke is obtained. However, when these char particles are crushed to eliminate the cenospheres and puffy structure, the coke is improved considerably.

Type of Pretreatment and Carbonization

The type of pretreatment and carbonization did not appear to have a significant effect on the properties of the formcoke when the design conditions were used. A few typical results for the sample pellets treated in different ways are listed in Table V. From the data obtained to date, the final temperature within narrow ranges (1750 to 1950) had only a slight effect on coke properties.

Evaluation of Coke Pellets for Metallurgical Use

The method developed to produce a good quality coke pellet consisted of pelletizing finely ground char by spraying a heavy residual oil binder on a disc pelletizer. These green pellets are combined with 1/2 by 1/4 inch coke medium and then cured on a grate with an oxygen rich gas to 500 F in 1-1/2 to 2 hours. The cured pellets are then screened from the medium and coked to about 1800 F in a vertical coking kiln in 2-1/2 hours. A flow diagram of the coke preparation step is illustrated in Figure 6.

Using these conditions, several hundred pounds of formcoke pellets were produced; a sample of the pellets is shown in Figure 7. The physical and chemical properties of these formcoke pellets were tested for comparison with metallurgical coke and another type of formcoke that had been used successfully in a blast furnace test. The test results are listed in Table VI. From these test data, it can be concluded that these formcoke pellets will make a good blast furnace coke. The good quality of coke obtained in the Clean-Coke Process results from 1) the fine pulverization of the char to break it down to the denser wall sections, 2) the use of sufficient quantities of binder to wet the solid particles, and 3) the intimate distribution of char and binder to obtain maximum strength. The photomicrographs in Figure 8 show the good microstructure of the formcoke and the good distribution of the materials.

A unique method has been developed to process char and binder derived from the process into a good quality formcoke. Attaining a high grade product enhances the overall potential of the Clean-Coke Process. Currently, information from process-development units is being used in the design of a pilot plant capable of processing four tons of coal per hour.

References

1. K. A. Schowalter and N. S. Boodman, "The Clean-Coke Process for Metallurgical Coke," *Chemical Engineering Progress*, Vol. 70, No. 6, June, 1974, pp. 76-82.
2. D. C. Rhys Jones, "Briquetting," *Chemistry of Coal Utilization*, Sys. Vol. H. H. Lowry Ed., John Wiley and Sons, 1963, pp. 675-753.

Table I

Selected Properties of Experimental Binders for Coke Pellets

	Asphalt		Coal-tar		Coal-		Fluxed		Pipe Line Enamelling Pitch
	AC 2000	RC 800	Pitch	CNBD*	Digestion Pitch	Coal Extract (De-ashed) Coal Blend)	CNBD	CNBD	
Asphaltene Content, wt%	1.4	1.6		CNBD*	CNBD		CNBD		NA
Viscosity @ 60 C poise	1900	634	167		Non- Newtonian	310			NA
Softening Point, Ring and Ball, C	49.4	14.8	34.2		84.0	42.3			51.5
Coking Value, wt %	19.4	17.6	36.4		29.4	29.9			NA
Benzene Insoluble	0.1	0.1	13.9		22.3	19.2			10.5
Quinoline Insoluble	0.0	0.4	6.5		CNBD	0.4			3.5
Carbon/Hydrogen Ratio	0.67	0.66	1.60		1.38	1.37			NA

* Could not be determined.

NA = Not available on sample used.

Table II

Pelletizing Conditions to Produce Green Pellets

Solids, 100% minus Size, 100 mesh	Char					
	1/8 in.	28-mesh	100 mesh	28 mesh	100 mesh	
% Char or Coke	60	60	60	60	57	
% Pitch Coke	13	10	7	11	9	
% Binder	27	30	33	29	34	
Type Binder	Coal- Tar Pitch	Coal- Tar Pitch	Coal- Tar Pitch	Asphalt (AC2000)	Fluxed Coal Extract	Coal Digested Pitch
Temp of Binder	360	360	365	350	370	370
Disc Angle, degrees	41	40	38	42	40	38
Disc Speed, rpm	30	30	30	30	30	30
% Recycle (Minus 3/4-inch material)	NA	20	NA	10	12	4
Green Pellet App Sp Gr	1.05	1.18	1.25	0.91	NA	NA

NA = Not Available

75-H-424 (008)

Table III

Effect of Binder Type on Strength of Resultant Coke Pellet

Type of Binder	Coal-Tar Pitch		Asphalt RE800		Coal Digested Pitch		Fluxed Coal Extract	
	28 mesh	100 mesh	28 mesh	100 mesh	28 mesh	100 mesh	28 mesh	100 mesh
Size of Char, 100% minus	1800	1800	1800	1800	1800	1800	1800	1800
Final Coke Temperature, F	700	950+	210	475	475	915	470	820
Crushing Strength, lbs (whole Pellet, 1-in.)	67	86	ND	ND	ND	ND	46	84
Tumbler Strength, Hardness (Plus 1/4-in., 700 rev)	0.87	0.94	ND	0.75	0.86	1.10	0.76	ND

ND - Not determined.

Table IV
Effect of Char Size on Strength of Resultant Coke Pellet*

Char Pulverized to 98-100% minus	<u>1/8-inch</u>	<u>28-mesh</u>	<u>65-mesh</u>	<u>100-mesh</u>
Crushing Strength, lbs (whole pellet)	590	700	630	820
Tumbler Strength (1400 rev) Plus 1/4-inch Index	53	59	64	74
Apparent Specific Gravity	0.94	0.97	0.88	1.10

* Pellets made from Illinois No. 6 seam char and coal-tar pitch. Carbonized in pilot test coke oven to 1800 F.

Table V

Effect of Method of Pretreating and Carbonizing on
Strength of Resultant Coke Pellet

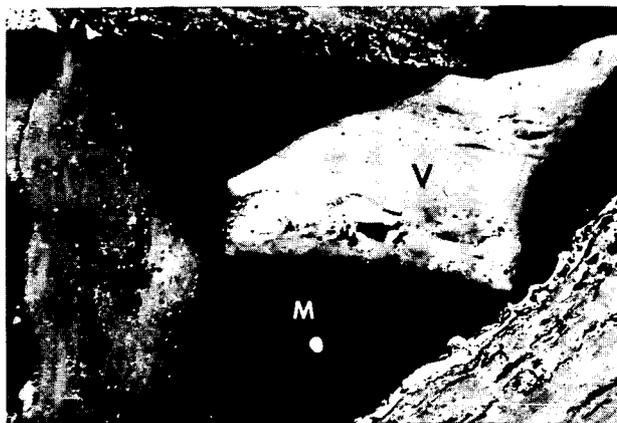
Type of Pretreatment	Type of Carbonization	Crushing Strength, lb (whole pellet)	Tumbler Strength Hardness*	Apparent Specific Gravity
A. Pellet Produced from Minus 28-mesh Char and Coal-tar Pitch				
1. Cured to 500 F (shallow bed at slow rate)	Pilot-Scale Coke Oven** (1800 F, no medium)	700	59	0.88
2. Preheated in Vertical Retort with Coke Medium to 900 F (Nitrogen Flow)	Pilot-Scale Coke Oven** (1800 F, no medium)	690	ND	0.81
3. None	Pilot-Scale Coke Oven** (with 1/8 in. x 0 coke fines, 1800 F)	750	59	ND
B. Pellets Produced from Minus 100-mesh Char and Coal-tar Pitch				
1. Cured to 500 F (Shallow Bed and slow rate)	Pilot-Scale Coke Oven** (1800 F, no medium)	835	74	1.13
2. Preheated in Vertical Retort with Coke Medium to 830 F	Pilot-Scale Coke Oven** (1800 F, no medium)	950	ND	1.10
3. None	Pilot Scale Coke Oven** (with 1/8 in. x 0 coke fines, 1800 F)	950	74	0.94
4. Preheated in Vertical Retort with Coke Medium to 830 F	Continuous Vertical Retort, 1800 F no medium	950	ND	1.10
* Plus 1/4-inch after 1400 rev.				
** Brennstoff Technik type coke oven.	75-H-424 (008)			80

Table VI
Properties of Formcoke

	Metallurgical Coke <u>1-1/4 x 3/4</u>	Coke Pellet <u>1-1/4 x 3/4</u>	FMC Corp. Formcoke <u>1-1/4 x 1</u>
Tumbler Strength*			
Hardness, plus 1/4 inch	85	85	83
Crushing Strength, lbs	600	>1000	810
Compressive Strength, psi	2450	2150	3170
Bulk Density, lbs/ft ³	29	32	32
Apparent Density	0.94	1.0	0.90
True Density	1.90	1.93	1.69
Porosity	50-1/2	48	47
<u>Chemical Analysis</u>			
V.M.	0.8	1.0	6.2
Fixed Carbon	92.2	91.5	87.1
Ash	7.0	7.5	6.7
Sulfur	0.7	0.5	0.75

* 22 lb sample for 700 rev in ASTM Tumbler.

ILLINOIS COAL



CHAR

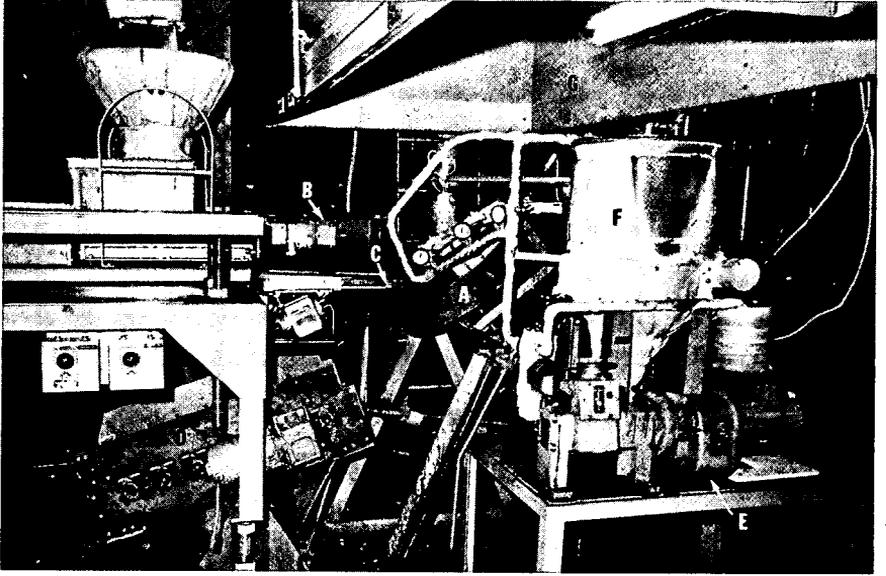


High-Volatile Char
Approximately 12% VM



Low-Volatile Char
Approximately 3% VM

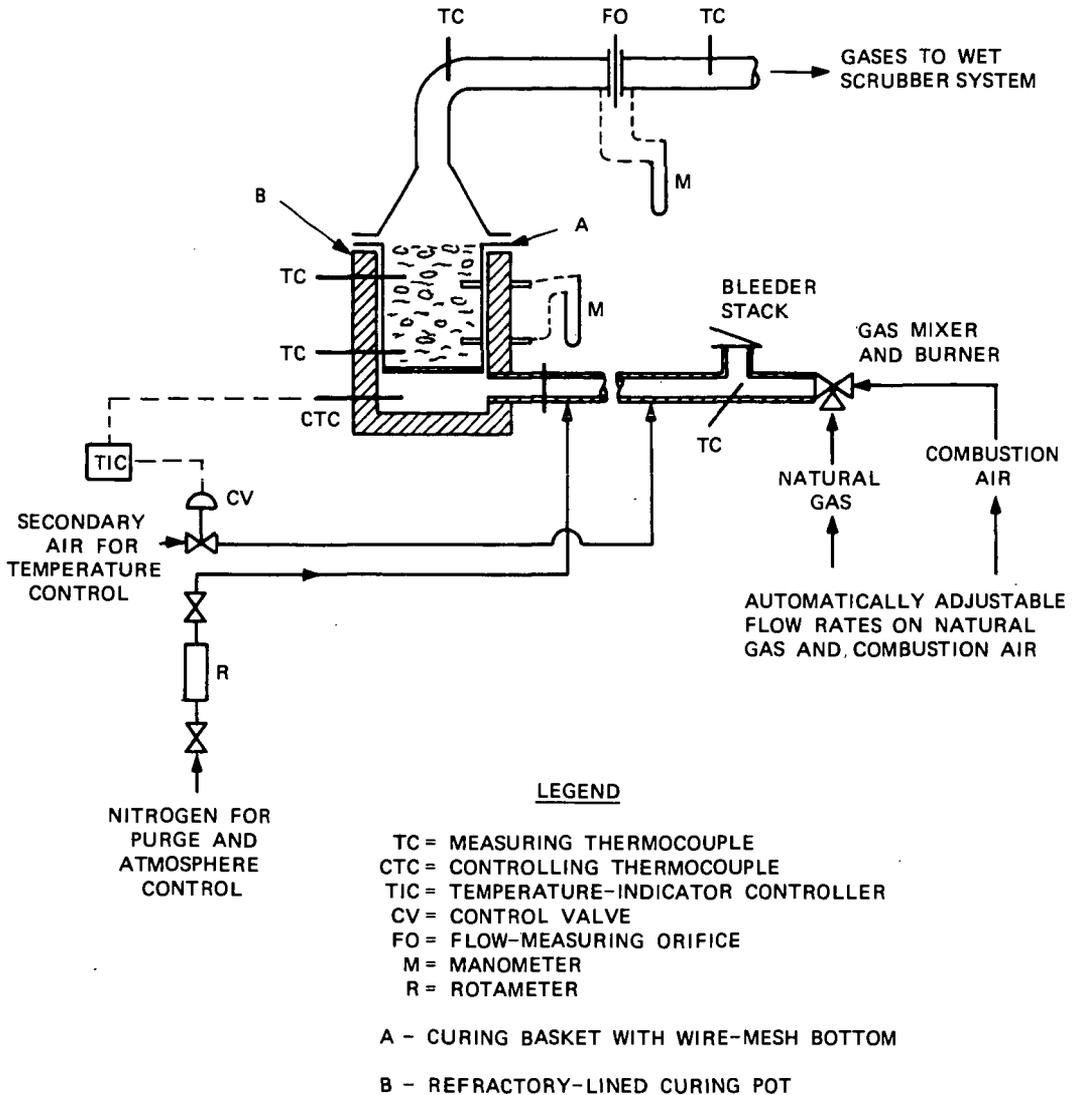
Photomicrographs showing typical appearance of Illinois No. 6 coal and the char product from coal which is used to make Formcoke pellets. (V) Vitrinoid (principal coal entity), (C) Char, (M) Mounting media, (P) Pores. Reflected light. X200



CHAR-PELLETIZING EQUIPMENT OF COKE-PREPARATION
PROCESS-DEVELOPMENT UNIT

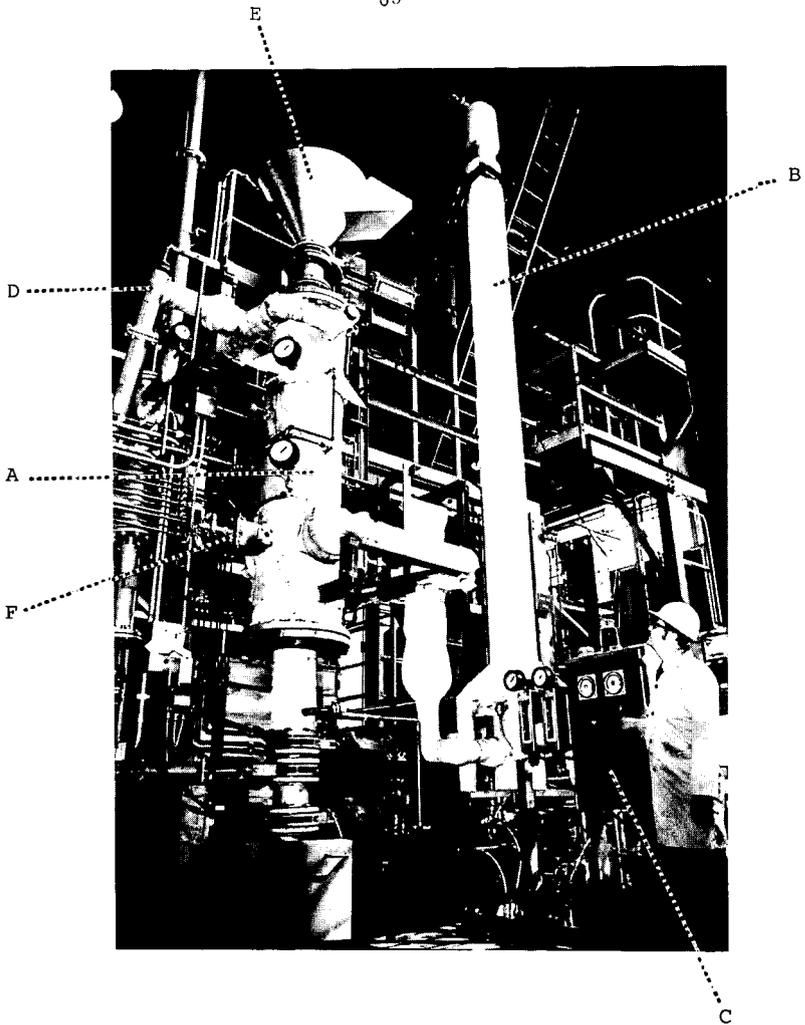
- A Two-Foot Pelletizing Disc
- B Char Screw Feeder
- C Char Distributor
- D Pellet Roller Screen
- E Binder Pump No. 1
- F Binder Surge Tank No. 1
- G Exhaust System

Figure 2



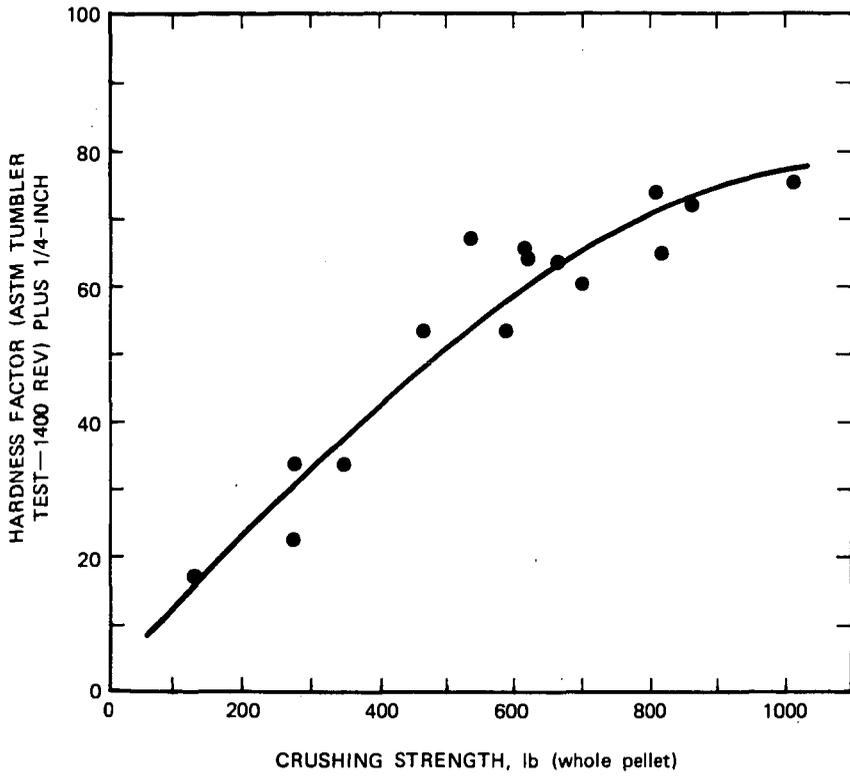
SCHEMATIC DIAGRAM OF CURING POT SYSTEM

Figure 3



Coking kiln and auxiliary equipment.

- A. Coking Kiln
- B. Kiln Gas Heater
- C. Heater Control Panel
- D. Kiln Gas Scrubber
- E. Pellet Surge Hopper
- F. Kiln Auxiliary Burner



RELATIONSHIP BETWEEN CRUSHING STRENGTH AND TUMBLER STRENGTH OF FORMCOKE PELLETS

Figure 5

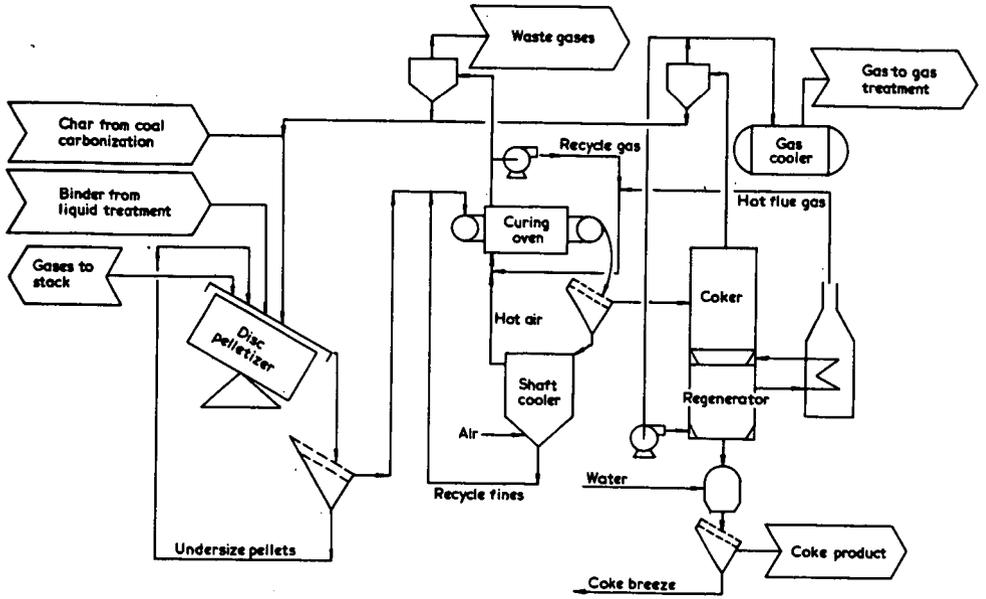
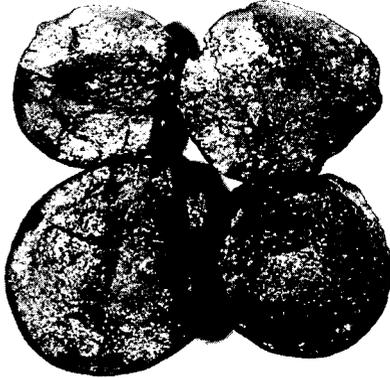


Fig 6. Illustration of equipment involved in the coke preparation during the Clean Coke Process

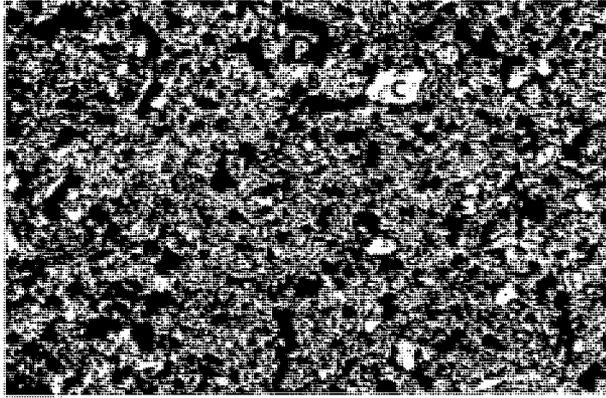


Broken Formcoke Pellets Showing Fracture Surfaces. X1.25

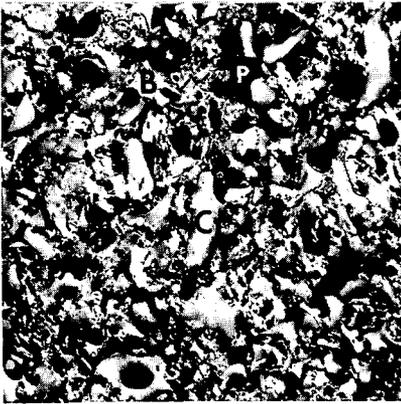


Formcoke Pellet Product

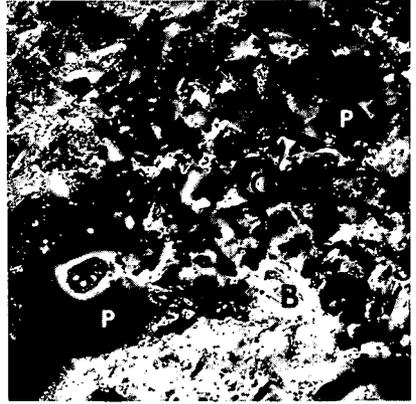
Photographs showing whole and broken of Formcoke pellets



Carbonized Formcoke Pellet Made From Minus 28-Mesh Char. X25



Good Mixing. X100



Poor Mixing. X100

Photomicrographs showing Formcoke pellet structure at low magnification and variation in microstructure due to mixing at high magnification. (C) Char, (B) Binder, (P) Pores. Reflected light.

THE MANUFACTURE OF METALLURGICAL FORMED COKE ACCORDING
TO THE BFL-HOT BRIQUETTING PROCESS

by

E. Ahland, J. Lehmann and W. Peters,
Bergbau-Forschung GmbH., Essen

and

J. Langhoff,
Ruhrkohle A.G., Essen

For the making of metallurgical coke, quite a number of continuous formed coke making processes have become known in the past years. It was the aim of these processes to produce lumpy coke suitable for blast-furnaces, above all from weakly or not caking coals. From a world-wide viewpoint, such coals are much more available than the prime coking coals from which conventional coke has been produced so far in slot-type recovery coke ovens.

Since 1962, Bergbau-Forschung GmbH., Essen, and the Lurgi Mineralöltechnik GmbH., Frankfurt, developed the so-called BFL-process which uses the hot briquetting of mixtures of coal and char in order to make formed coke. For the development of such a process for the thermal refining of hard coal, several years of testing and trial are indispensable. Subject of such a phase of trial are the process as such as well as the products made according to this process.

As for the trial and the process itself, the question arises first of all about the technical feasibility of the process. When this feasibility has been principally proven, questions of a suitable selection and set-up of machines and apparatus gained in importance, while the optimization of the overall process concludes the development. The necessary tests can be carried out in small-scale testing or trial plants or with physically similar models. Both methods of development are suitable and complement each other meaningfully. In this connexion, the question of the transferability of the results, first from small-scale plants to large-scale plants, and then, in case of model tests from the model to the large-scale realization, has an essential importance.

It is the objective of a testing and trial of the products to prove that the new product is at least equivalent to those hitherto used. A two-way procedure offers itself also for this stage of trial: on the one hand, trials can be run in small-scale plants as are available, e.g., to the West European metallurgical industry in the trial blast-furnace Ougrée, or, on the other hand, prediction about the behaviour of the product in practice can be made from the chemical, physical and technological properties obtained from the laboratory tests. It is a matter of course that - in order to establish the evidence of the formed coke for its suitability - a long-term trial in a practical blast-furnace for at least one month will be necessary. Without such a long-term trial nobody will risk to establish a commercial plant according to a new process, as the investments required therefor would be too high.

The BFL-process¹⁾²⁾ has already passed a considerable part of the lead time up to the unlimited application of the process in practice. For better understanding of the following expositions, however, the principle of the BFL-process will be described once more below.

Description of the process

As shown in Figure 1, hot and finely grained char with a temperature of about 750 °C is mixed with finely grained binder coal in a mechanical double screw mixer. The binder coal should be available in a size <1 mm. The ratio between binder coal and char is about 30:70. On account of this ratio and the temperature of the hot char, the temperature of the mixture is in the range of softening of the binder coal. The double screw mixer has the function to mix rapidly and intensively the char and the binder coal in order to attain a balance of temperature between the two flows at the end of the mixer. Thus, the binder coal has been heated up to a temperature of 450 °C and has become plastic. The mixture then gets into a pug mill from where it is conveyed to the double-roll press. This press gives the briquettes their shape. The briquettes leaving the press at a temperature of about 450 °C must be subjected to a careful cooling and are then eligible for use in the blast-furnace.

As Figure 2 shows, there are two methods for producing the necessary char. The more simple process and also that requiring less investments is the fluidized-bed carbonizer. Here, the fine coal fed into the reactor at a temperature of about 750 °C is carbonized in the fluidized bed. Part of the necessary heat can be supplied with the fluidizing gases, another part by the burning of char in the fluidized bed. The oxygen necessary for combustion is contained in the

fluidizing gases. The waste gas consists of a mixture of degasification gas and the fluidizing gases. Due to its high nitrogen contents, however, it has still only a low calorific value in the range of 1000 to 2000 kcal/m_n³.

The more expensive process circulating a heat carrier according to Lurgi-Ruhrgas, briefly called "the LR process", provides a separation of the heating-up process and of the degasification process with the result that a rich gas is obtained as a by-product. Circulating char at a temperature of about 750 °C and fine coal, in a ratio of 10:1, are supplied to a mechanical double-screw mixer. This mixture is then brought, with a temperature of 650 to 700 °C, into a subsequent degasifier from which the product gas on the one hand can be withdrawn and, on the other, the excess fine char. The excess fine char is supplied to the hot briquetting section, while most of the char remains in the circulation and is supplied to an airlift. In this airlift the char now used as a heat carrier is re-heated up to 750 °C either by partial combustion or by a bottom firing with fuel gas. In the collecting bin, the char is separated from the carrier gases and re-enters the process, while the flue gas, with its content of sensible heat, can be used for pre-heating the combustion air for the airlift and the drying of coal. The selection of the process for the production of a finely grained char is not without influence on the properties of the product.

The LR-process yields, as a whole, a denser char and, thus, also denser hot briquettes. Generally, all hard coals, from the anthracite to the long-flame coal, are suitable for char making. The binder coal, however, must fulfill certain requirements, viz. concerning the coking and the softening properties. Recent investigations in the laboratory and in our semi-technical plant showed that not only well-caking coal but also medium and weakly caking coal with a Swelling Index of ca. 3 can be used successfully as binder coal, provided part of the tar obtained during low-temperature carbonization and hot briquetting is admixed to the hot briquetting mixture. By adding about 6 to 9 % of tar to this mixture, the binding capacity of the coal is improved to such an extent that hot briquetting strengths are reached as with the sole use of a well caking coal as binder component.

Testing and trial of the process

The process has, so far, been tested and tried in four stages. Parallel to the investigations in the testing plants, individual problems of the development of the process were solved in a number of model tests. After reaching the next higher stage, however, the smaller-scale testing plants did not usually become superfluous as the duty to investigate several

times new coals necessitated each time tests and trials in the smaller-scale apparatus. Besides, the process itself is being further developed and improved continuously, as, e.g., now weakly or medium caking coals can be used as binder coal with the addition of tar or other binding agents to the hot briquetting mixture.

The smallest-scale plant, as shown in Figure 3, is a laboratory apparatus. This is, in fact, a discontinuously operated briquetting plant working with a small piston press. This press yields briquettes with a weight of about 12 g. From density and strength investigations a first statement can be made about the suitability of the used coals as well as about the presumably best operating conditions. In small coking ovens, the behaviour of these green briquettes during the subsequent heating-up can be investigated.

The next stage involves semi-technical plants with a throughput of 100 to 600 kg/h. These plants are working continuously with a double-roll press. For the production of char, an LR-plant as well as a fluidized-bed carbonizer are available. Figure 4 shows the building accommodating these plants. But even testing plants of this size do not yet permit to produce the quantities required for blast-furnace tests. This became possible only by a plant with a throughput of 5 t/h of hot briquettes shown in Figure 5. In this plant, quantities could be produced for blast-furnace tests with throughputs up to 7000 tons per test.

The presently last step of development are the large-scale testing plants. Messrs. Still of Recklinghausen established, by order of the Ruhrkohle AG, during 1973 and 1974 a 12.5 t/h hot briquetting plant on the area of the coke oven plant Prosper in Bottrop. This plant is operated by the "Arbeitsgemeinschaft Formkoks" formed by experts of Ruhrkohle AG and of Bergwerksverband GmbH., a subsidiary of the Bergbau-Forschung GmbH. Figure 6 will give you an impression of this plant which, compared with conventional coke oven plants, has been built much more to the height.

A further large-scale testing plant according to the BFL-process with an throughput per hour of 27 tons of hot briquettes is being built at present, by order of the British Steel Corporation, by the Lurgi Mineralöltechnik GmbH. in England.

These large-scale testing plants are to fulfil three duties:

1. Testing of the BFL-technology in a larger scale.
2. Making sufficient quantities of hot briquettes available for long-term blast-furnace tests.
3. Drawing-up of reliable material and heat balances to serve as a basis for economy calculations.

The flowsheet of the large-scale testing plant Prosper and its individual stages: drying, LR-low-temperature carbonization, hot briquetting, the cooling of briquettes and the purification of gases, can be seen from Figure 7.

The coals delivered by rail are stored separately, according to coal for char making and binder coal and are then dried after withdrawing through vibrating chutes into two parallel flash driers (1). The about 700 °C hot waste gases of the LR plant are used for drying. Afterwards, the moisture content of the coal is < 1 %.

The dried binder coal is crushed in an impact mill (2) to < 1 mm. As for the coal for char making, the sizes > 3 mm separated in a pneumatic deduster can also be subjected to crushing (2). According to our experience, however, crushing can be resigned on because of a sufficient crushing of the charring coal particles during the shock type heating-up during carbonization. The dried coal is bunkered for a while in the plant. The char is produced according to the LR-process. For this process, the coal for char making is proportioned, in a ratio of 1:10, to the circulating char, via a belt scale, in the double-screw mixer. By the following admixture of fresh coal, the temperature of the mixture decreases to about 700 °C. After a short residence in the degasifier shaft, the mixture is proportioned to the airlift to which the heat necessary for low-temperature carbonization is supplied, either by the combustion of char or the combustion of gas. In the ascension pipe, the char is heated up to 750 °C and conveyed into the collecting bin. The bulk of the char is returned to the circuit and, thus, into the mixer, while the excess char flows into the hot briquetting.

The waste gases of the airlift are passed, via a cyclone system (4), into a secondary combustion chamber where the residual coke dust and the combustible gases are burnt. The heat of the waste gases is utilized for the pre-heating of the air to the airlift and in the driers.

For hot briquetting, the binder coal is proportioned, via a belt scale, to the double-screw briquetting mixer (5) and is there intensively mixed with the hot char. In the subsequent pug mill (6) the final homogenization and degasification of the briquetting material take place which flows to the roll press by gravity (7).

The used briquetting rolls have a diameter of 1.4 m and a maximum contact pressure of 3.5 t/cm. Briquette sizes of 20 to 300 cm³ can be produced.

Directly below the press, the grooves are separated from the briquettes by a fixed grate and are returned into the LR-plant.

The hot briquettes are cooled in a shaft cooler (8) by circulating gases, these gases being cooled themselves in a warm water cooler in order to avoid a condensing of water. At the end, the briquettes pass a water bath where the residual heat is discharged.

The carbonization gases from the LR-plant and the hot briquetting are subjected for purification to a several-stage condensing process where tar, oil and gas liquor from the carbonization are obtained, in addition to the gas.

As concerns environmental protection, the hot briquetting process can be considered as "non-polluting". All stages, from drying up to the cooling of the products, take place in closed containers and apparatus. Contrary to the presses in conventional briquetting processes, the hot briquetting press, too, is totally encased. This, however, hinders the attendance and the operating of the press by no means.

The dedusting of the waste gases from the drier and the collecting bin of the LR-plant takes place in an electrostatic precipitator down to a value - prescribed by the Mines Inspectorates - of 150 mg/m^3 , after the residual CO and H_2 have been burnt in a combustion chamber.

Desulphurized carbonization gas can be used for bottom firing of the LR-plant with the result that the dust gases, too, are free of sulphur.

All gases and water flows containing any contaminations are fully under control. As regards measuring and control, the entire plant has been conceived such as to be controlled from a central control room. No personnel needs to be present in the station, except for repair and attendance work. Thus, the demands placed on a modern hygiene at the working point have been realized to a wide extent.

The taking into operation of the demonstration plant Prosper began in May 1974 and has not yet been completed up today. Figure 8 containing schematically the individual sections with the ancillary plants, shows (in a dark colour) which components of the plant have been taken into operation so far.

A number of technical disturbances and standstills - not unusual for commissioning such a demonstration plant - caused considerable delays in the originally planned time schedule. Deficiencies in the conventional equipments formed a frequent source of defects and failures. E.g., the drying and re-heating phase of the refractory material required a much longer time as unexpected conditions of the Inspectorates with regard to the safety in operation had to be observed.

At the time when this report was written, the LR-cycling had been taken into operation and was well under control. The time schedule provides as a next step the taking into operation of the gas purification, which will be followed by the commissioning of the hot briquetting stage.

Testing of and trials with the products

A first indication on the suitability of different formed cokes for use in blast-furnaces is obtained from the data of analyses. The interesting properties are: size and shape, strength, density, reactivity and contents of volatile matter. These factors are much influenced by the control of the process, while other factors, such as the ash and sulphur contents, depend essentially on the type of input coals.

The size and shape of the briquettes can be freely selected during the briquetting process, the shape being subjected to a more stringent limitation than the size. In the 5 t/h plant, hot briquettes have been produced up to a maximum of 300 cm³. This large briquette, however, was not used in blast-furnaces, but in cupolas. Thorough model investigations about the flow resistance of formed coke led to new perceptions. Of decisive influence on the strength of burdens of formed coke is the purling of the burden into the void space of the formed coke.

For the lower part of the blast-furnace, e.g., the zone where the iron has been molten yet, a formed coke as lumpy as possible, is desirable in order to attain a low flow resistance. On the other hand, a lumpy coke can be disadvantageous before the tuyeres as well as in the shaft of the blast-furnace. In front of the tuyeres, the surface offered can be so low that the turn-over slows down with the blast. In the upper part of the blast-furnace, the finely grained sinter can purl into the void space to such an extent that the pressure loss will be higher than with small sizes of coke. Thus, there is an optimum size which always depends on the type of burden used. Generally, the optimum size is smaller when pellets are used than with the use of finely grained sinter. This know-how obtained first in model tests could be much confirmed in actual blast-furnace trials.

The strength of the formed coke plays an important part during trials in the blast-furnace. In order to be able to draw a comparison with the conventional coke, the drum test offers itself, as the crushing strength usual for briquettes cannot be applied to a conventional coke, because of the irregularity of the coke lumps. Figure 9 shows - besides other properties - some comparison strengths according to MICUM as well as according to IRSID. Attention must be paid, however, that the abrasion < 10 mm developing from formed coke is finer as when conventional coke is subjected to drum tests.

After the trials by Ledent⁴⁾ in the Ougrée blast-furnace, the attention has been directed to the coke density and the porosity connected therewith. According to Ledent, formed coke should have, if possible, a similarly high porosity as conventional coke. An extremely dense coke is not suitable for use in a blast-furnace.

The reactivity of formed coke is, as a rule, higher than that of conventional coke. However, it is still within a range where it is without much influence on the operation of the blast-furnace.

Important in connexion with the behaviour of the hot briquettes in the blast-furnace was the question whether the burn-up of the green briquettes takes place from the outside or through the pore system in the inside of the green briquettes. In the latter case, the burn-up in the blast-furnace could have caused a decisive weakening of the green briquettes with the result that they could fall to pieces in the bottom part of the blast-furnace. Samples drawn from the tuyeres during blast-furnace tests showed, however, that the burn-up takes place wellnigh exclusively via the outside surface of the briquette and that the structure in the rest of the briquette remains nearly unchanged.

When using uncoked hot briquettes in the blast-furnace, a much higher content of volatile matter must be reckoned with as in the case of conventional coke, as is to be seen from Figure 9. As far as these volatile matters emit from the briquettes in form of gas, they will not interfere with the operation of the blast-furnace. The blast-furnace gas will be enriched thereby in its calorific value what was not even unwelcome in many of the works. The volatile matter must not contain any traces of tar, as otherwise operational defects could be caused by its condensation in the equipment subsequent to the blast-furnace. Tests run so far, however, gave not reason to such fears.

That this must not be expected from longer-lasting tests has been shown by comparable tests where steam coal nuts

(with 17.5 % Vol. Matter waf) were added to the coke (to the extent of 6:8 %) in operating blast-furnaces of the Youngstown Sheet and Tube Company); even after an operation of one year no depositions could be found in the subsequent pipelines.

Figure 10 gives a review of the blast-furnace tests and trials carried out in the meantime with BFL-formed coke. Green as well as calcined briquettes were tried, the hot briquettes being charged to the extent of 100 %, i.e. without admixing conventional coke. The strength of the hot briquettes proved to be sufficient for the transport processes outside the blast-furnace as well as for the mechanical stresses inside the blast-furnace. No breaking of the briquettes could hardly be observed. Dust development in the transport cars in front of the blast-furnace remained low. In the blast-furnace gas as well, the dust contents increased only a little, compared with the operation with conventional coke.

The tests and trials covered the period 1967 to 1973. They were run in Belgium, the Federal Republic of Germany and in England. The diameters of the hearth-casing of a blast-furnace reached from 1.4 m to 9.5 m, and the period of testing from one day to seven days. After the demonstration plant Prosper has been successfully taken into operation, it is provided to prove the definite suitability of the new fuel in blast-furnaces in long-term trials.

To close with, the advantages of the BFL-process will be summarized once more:

The process permits the use of coals otherwise not suitable for coking, thus enlarging essentially the coal basis for making coke.

The process can be considered to be "non-polluting", as the coking process takes place in closed apparatus from which the developing flows of waste gas can be got hold of fully and can be purified by conventional methods.

The entire plant can be started and stopped quickly. Thus, the process is most flexible and can be easily adjusted, if necessary, to a varying market for coke.

Thanks to the free selection in size and shape, the hot briquettes can be adjusted to different types of burdens with a view to a good permeability across the blast-furnace.

References

- 1) Ahland, E., J. Lehmann and W. Peters: Suitability of Coal for Production of Formed Coke. 31. Ironmaking Conference, Chicago 1972.
- 2) Schmalfeld, P. and R. Rammner: Results of the Development and Present Stage of the BFL-Hotbriquetting Process. 13th Biennial Conference of the Institute for Briquetting and Agglomeration, Colorado Springs, August 1973.
- 3) Schultz, H.-J. and O. Abel: Druckverluste in Koks-Sinter-Schichtungen und -Mischungen. Arch. Eisenhüttenwesen 45 (1974) No. 7, pp. 445-448.
- 4) Ledent, P., G. Burton and M. Marcourt: Die Entwicklung des INIEX-Verfahrens zur Erzeugung von Formkoks. Informationstagung der EGKS, Luxemburg, April 1970.
- 5) Harris, W. F., R. J. Tracey and D. L. Locier. Substitution of Coal for Coke in Blast Furnace Burdens. Paper presented at Eastern States Blast Furnace and Coke Oven Association, Pittsburgh, Pa., February 9, 1973.
- 6) Därmann, O., S. Henkel and K. D. Haverkamp: Die Verwendung von Heißbriketts und Formkoks im Hochofen. Stahl und Eisen (1970) pp. 1009-1012.
- 7) Holgate, J. K. and P. H. Pinchbeck: Use of Formed Coke: BSC Experience 1971/1972. J. of the Iron and Steel Institute 1973, pp. 547-566.

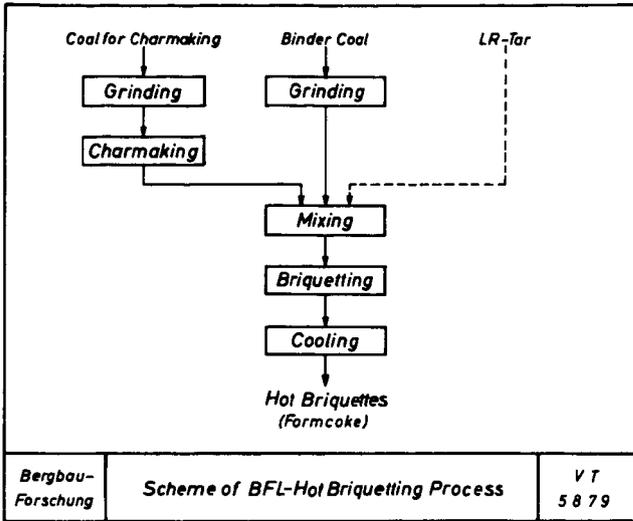


Figure 1

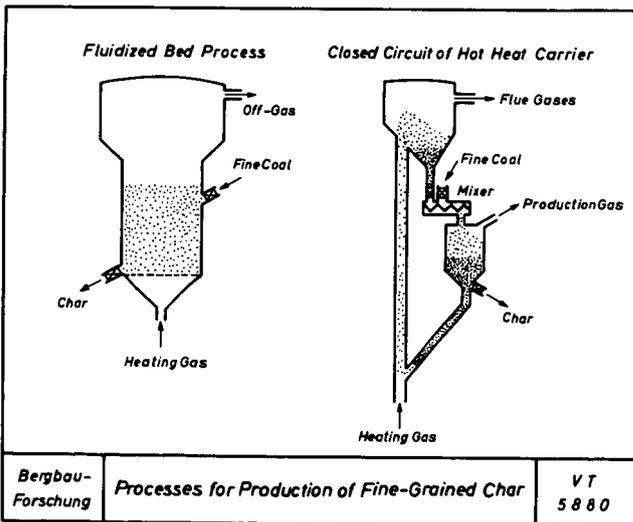


Figure 2

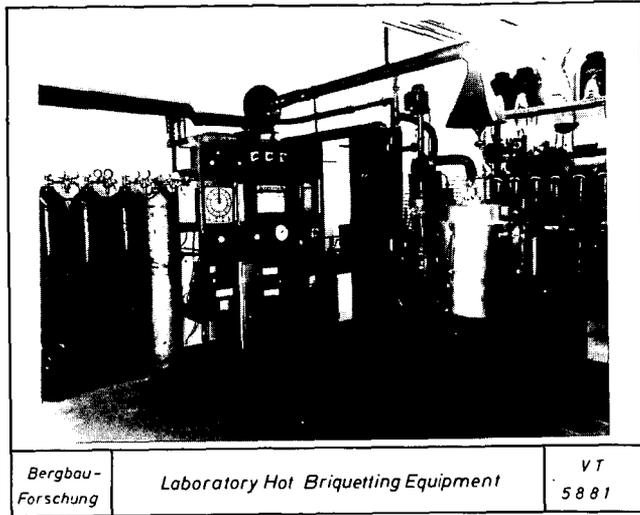


Figure 3

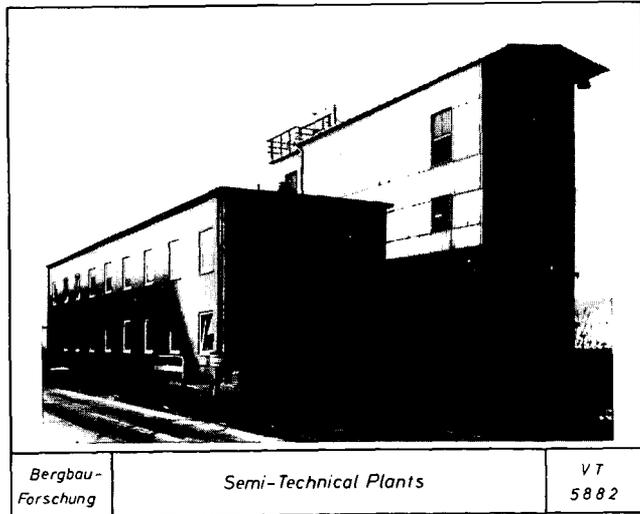


Figure 4

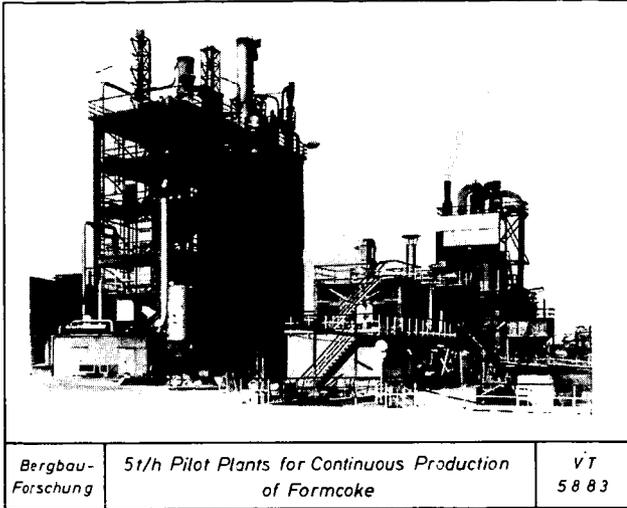


Figure 5

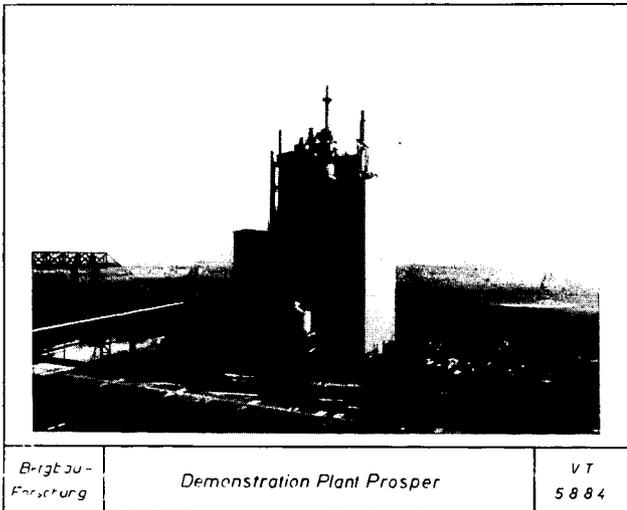


Figure 6

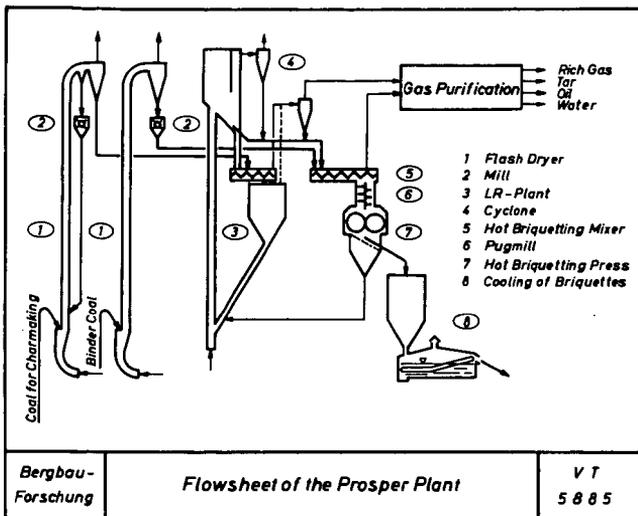


Figure 7

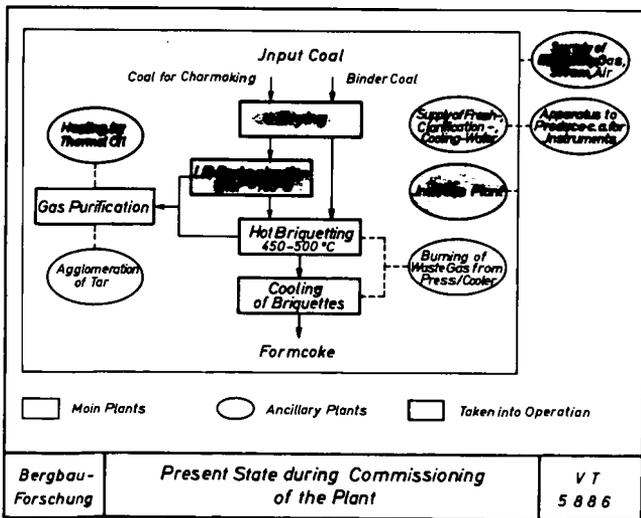


Figure 8

	BFL-HOT BRIQUETTES		BF COKE III
Weight per Piece.....g	101	48	—
Porosity.....%	39	49	50
Vol. Matter.....%	10,1	7,1	< 1
Ash.....%	6,8	7,0	8,6
Sulphur.....%	0,91	0,83	0,86
Bulk-Density.....Kg/m ³	561	508	437
STRENGTH			
MICUM [DIN 51717]			
M ₄₀%	92,6	91,7	81,2
M ₁₀%	6,7	7,8	6,5
IRSID			
I ₂₀%	79,0		80
I ₁₀%	20,8		18
BF 1975	PROPERTIES OF BFL-HOT BRIQUETTES AND CONVENTIONAL COKE		5887

Figure 9

TEST	1	2	3	4	5	6
Year	1967	1969	1970	1970	1971	1973
Country	BEL	BRD	BRD	BRD	GB	BRD
Dio of Hearth-Casing..m	1,4	9,5	6,8	6,8	5,5	6,8
Tonnage.....t	500	1350	2700	7000	2650	1150
PROPERTIES OF BRIQUETTES						
Weight.....g	21	45	48	44	53	98
Volume.....cm ³	26	49	55	51	58	106
ABRASION M10						
[DIN 51717].....%	6,7	7,0	7,8	7,1	12	10,4
Porosity.....%	57	54	49	59	43	45
Bulk-Density.....Kg/m ³	415	558	508	495	507	495
Vol.-Matter.....%	2,5	1,9	7,1	1,7	6,0	9,2
BF 1975	BLAST FURNACE TESTS WITH HOT BRIQUETTES					5888

Figure 10