DOMESTIC COKE FROM ILLINOIS COALS
AN EXPERIMENTAL SOLE-HEATED OVEN

BY
FRANK H. REED AND HAROLD W. JACKMAN

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DOMESTIC COKE FROM ILLINOIS COALS
AN EXPERIMENTAL SOLE-HEATED OVEN*

By
FRANK H. REED AND HAROLD W. JACKMAN

ABSTRACT

The immense reserve of bituminous coal in Illinois and the growing demand for
smokeless domestic fuel have caused the Illinois State Geological Survey to extend
its research on coal carbonization. Previous small-scale studies have shown that
domestic coke can be made from many Illinois coals. The present research pro-
gram includes carbonizing studies in experimental pilot-size ovens on coals from
the various mining districts of the state. An oven of the sole-heated type has been
built which is capable of carbonizing 350 pounds of coal under conditions appro-
aching commercial practice. Electric heating is used to obtain accurate temperature
control. Equipment is provided for gas purification and for by-product recovery.
Coking studies on this oven with coals used in commercial ovens of similar design
show close correlation between experimental and commercial yields, and also in
quality of coke, gas, and tar.

ILLINOIS has larger reserves of high-volatile bituminous coal than any other state.
Nearly three quarters of the state is underlain by coal. The reserves are estimated at
approximately 150 billion tons. If production of coal in Illinois were maintained at
50 million tons per year, which is an average rate for the past few years, and if 50% of
the coal were recovered, the reserves would last 1500 years. These coals are principally
high-volatile bituminous B and C coals, according to the A.S.T.M. system of classifi-
cation by rank (1, 3). They are lower in rank and weather more rapidly than the
Appalachian coals of Pennsylvania, West Virginia, and eastern Kentucky; Illinois
coals which have been stocked over an ex-
tended period show decided deterioration in coking properties.

Serious attention was first given to the
coking of Illinois coals from 1902 to 1927
by Parr (8, 9, 11-22). A low-temperature
carbonization process was developed and a
small plant operated for one year. This
plant was owned by private interests and
there are no public records, but it is known
that coke was produced. The process has
never been commercialized. Further study
was undertaken from 1932 to 1937 by
Thiessen (23, 24). Coals from fifteen
mines in Illinois were tested in the U. S.
Steel carbonization assay equipment (26)
and in 6-inch cylindrical retorts. Results
showed that, under favorable conditions,
coke suitable for domestic fuel could be
made from many of these coals.

Another development during this period
was the commercial Knowles-Curran type
of coke oven (2, 5, 6, 7). Coke plants
equipped with oven batteries of this type
were built at West Frankfort and later at
Millstadt, Ill., for the primary purpose of
producing domestic coke for the St. Louis
market. These plants carbonize southern
Illinois coal screenings. Acceptable domestic
coke has been produced and sold continu-
ously since 1934.

In 1940 and 1941 the State Geological
Survey built an industrial laboratory which
provided space for a small-scale experi-
mental coke oven. A research program was
planned to evaluate Illinois coals for the
production of smokeless domestic fuel ac-
ceptable in the city of St. Louis and other
communities having smoke abatement ordi-
nances.

The program includes studies with the
U. S. Steel carbonization assay equipment,
followed by processing of coals from the
various mining districts of the state in ex-
perimental ovens designed to reproduce
carbonizing conditions in present commer-
cial ovens. Gas purification and by-product
recovery are provided which make possible
a material balance on the products of car-
bonization. By such a program Illinois coals

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try at the 106th Meeting of the AMERICAN CHEMICAL
SOCIETY, Pittsburgh, Pa.
may be carbonized and evaluated, either individually or blended with one another or with other coals. The effect of coal preparation, petrographic analysis, size consist, weathering, and other factors may also be studied.

As a preliminary guide to investigating the coking properties of Illinois coals, the U. S. Steel assay test (26) was run on some fifty coals and coal blends. Test results were found to be accurate and to indicate proper procedures for pilot plant investigation. This small-scale unit gave coke and by-product yields comparable to commercial practice. Blending up to 30% eastern low-volatile coal with Illinois coals improved the physical characteristics of the coke. This unit was also used to investigate the coking properties of the banded ingredients of Illinois coals (4, 25) and showed decidedly different coke structures from the various coal bands. The assay test showed certain coals to be good prospects for semicommercial investigation and probably for commercial carbonization.

Following this preliminary investigation, a pilot oven of the sole-flue heated type was constructed, capable of carbonizing up to 350 pounds of coal under conditions approaching commercial practice. This oven
was provided with condensing equipment for recovery of by-products and with apparatus for determining the heating value and quantity of coal gas. The oven has been under heat since November 1942, and forty runs have been made. No major operating trouble has been encountered, and close correlation between experimental results and commercial results in similar equipment has been noted.

The front view of the sole-heated oven with coke ready to pull is shown on page 6. Its construction (Figure 2) follows closely that of the Knowles-Curran ovens except that it is heated electrically in order to permit close temperature control which is essential to an experimental oven of this type.

DESCRIPTION OF OVEN

As in the commercial ovens, all heat is applied from flues below the oven floor. This oven has been provided with three such heating flues, 1 (Figure 2); each is approximately 9½ inches wide and extends the full length of the oven. Three hairpin coils of Kanthal A wire, 2, in each flue are supported on an assembly of insulating firebrick, 3, set on a transite plate, 4, and placed in the flue so that the coils are directly below the oven chamber. These insulating brick are grooved to fit the coils, and the grooves are lined with half sections of aluminum tubes. The coils, ¾ inch in diameter, are placed so as to leave a clearance below the oven floor of ½ inch. There are two layers of insulating firebrick, 5, in the base of the oven so that little heat is lost below the coils.

Oven floor, 6, is constructed of silicon carbide tile, 1½ inches thick. These floor tiles are not cemented to the brick on which they rest, and are free to expand and contract with temperature change. They may be removed and replaced in case of breakage. Substitution of silicon carbide tile for the usual silica floor tile reduces the heat gradient across the floor and ensures more even heat distribution. Therefore electric coils that give a flue temperature of 2300°F provide adequate floor temperatures.

The carbonizing chamber, 7, is 34 inches wide, 36 inches deep, 26 inches high at the sides, and 33 inches high at the top of the arch. The ratio between the space occupied by the coal and the free space above the coal is approximately the same as that in the commercial ovens. The side walls are 19 inches thick and insulated by embedded vermiculite insulating brick. The arch is made of standard arch brick, covered with a layer of vermiculite insulation and finally with common red brick to form a level operating floor. In the center of the arch a hole 9 inches square is provided, 8, through which coal is charged into the oven; a plug of refractory concrete closes this charge hole during carbonization. The back of the oven is permanent brickwork, and the front is left open. A door of refractory concrete, 9, which covers the opening, is hung by a cable and counter weight for quick raising and lowering. A temporary brick wall, 10, either three or four courses high, depending on the depth of coal charge to be used, is set without mortar just inside the oven door. When charged into the oven, coal is leveled and retained against these brick, and the brick are removed when carbonization is completed.

The entire oven structure is held together by tie rods, 11, extending through the top brickwork and through the oven foundation, and anchored to heavy angle-iron back stays, 12, at each corner of the oven. Steel angles are embedded in the side walls, 13, and placed where they will support any side thrust from the arch. With this thorough steel bracing there has been no tendency for cracks to develop in the oven brickwork.

Temperatures inside the oven chamber are shown by thermocouples inserted through the side wall—one at floor level, one in the center of the coal charge, and one in the gas chamber at the base of the arch, 14. Temperatures are registered by a four-point recorder and give a graphic record of the progress of temperature change through the coal charge.

CONDENSING EQUIPMENT AND BY-PRODUCT RECOVERY

The gaseous products from carbonization are cooled and are scrubbed to remove tar, ammonia, hydrogen sulfide, and, when desired, light oils. The quantity of ammonia removed is not determined. Light oil removal, although provided, has not been included in the correlation studies because commercial plants of this type do not have oil scrubbers. The purified gas is metered,
Fig. 2.—Sole-Flue Coke Oven, Electrically Heated
and a continuous record is made of its heating value throughout the carbonization period. A composite gas sample is collected in a water-seal gas holder by adding proportional increments of gas to the holder each half hour. At the end of the coking period this composite gas sample can be used for determining heating value or gravity, or for chemical analysis.

The units which make up the condensing and by-product recovery equipment are shown in Figure 3. Raw gas from oven A is first washed by a liquor spray at point B, just beyond the oven outlet. Gas then enters the base of washer-cooler C, where it is further washed and cooled and where the bulk of the tar is removed. C is a 6-foot length of 12-inch-diameter pipe, packed with 3-inch spiral tile and having a liquor spray at the top. Pump P3 circulates washing liquor to both B and C. This liquor, containing tar scrubbed from the gas, returns by gravity to tar-liquor separator D, where the tar settles out and the liquor is recirculated. D is a 75-gallon tank built with tar baffles; it has a false bottom under which tar collects and from which it can be drained.

Leaving the washer-cooler at about 110°F., gas passes through tar scrubber E, where it is further cooled to 65°F. and where the remaining tar oils are removed. In this unit, water at city pressure is fogged into the gas stream and subsequently removed by baffles, carrying with it the tar oils which are separated from the water in separator F and later combined with the bulk of the tar in D. Both separators, D and F, overflow into waste liquor tank G which, in turn, overflows into the sewer.

From the tar scrubber the gas is pumped by a Roots-Connersville exhauster, H, into hydrogen sulfide scrubber I. I is a 7-foot length of 12-inch pipe, packed with 1-inch Berl saddles and having a spray at the top. Pump P2 circulates a concentrated solution of soda ash through the scrubber, removing from 90 to 100% of the hydrogen sulfide from the gas. Any remaining hydrogen sulfide is removed in an oxide catch box, J, constructed from an oil drum and filled with a commercial brand of iron oxide sponge. The amount of hydrogen sulfide present at any point in the purification system may be determined by a Tutwiler apparatus (10).

Following this step of purification, the gas passes through light oil scrubber K, which is a 10-foot length of 12-inch pipe packed with ½-inch Berl saddles through which absorbing oil can be circulated by pump P5.

Beyond this point the gas is completely purified and is metered by a diaphragm-type gas meter, L, having a large registering dial. Following the meter a portion of gas
AN EXPERIMENTAL

goes to gas calorimeter $M$, another portion is retained at half-hour intervals in gas holder $N$ as mentioned before, and the balance is exhausted to the atmosphere. $M$ is a Cutler-Hammer Thermometer which both indicates and records total British thermal units continuously. $N$ is a gas holder of 5-cubic-foot capacity, equipped with an indicating scale which makes possible the accurate addition of small increments of gas.

If desired, each unit in the condensing train following the exhauster can be valved off and by-passed. Provision is made also for inserting additional units at any point in the system. Pressure gages and thermometer wells are provided at numerous points throughout the train so that the operation of each unit can be noted and controlled.

OPERATION OF OVEN

In operating the small coke oven and auxiliary condensing equipment, various oven flue temperatures and depths of coal bed have been tried. Results most nearly duplicating commercial practice have been obtained at a flue temperature of 2200°F, and a coal depth of 7½ inches. Under these conditions the coke bed just above the oven floor is heated to 1800-1900°F. Complete carbonization requires from 6½ to 10 hours, depending upon factors such as coal moisture content, bulk density, and plastic characteristics of the coal.

In the operation of a commercial coke oven battery, the heats once attained remain fairly constant, and fresh coal is charged into an oven kept at approximately the same temperature at which coke was previously pushed from the oven. In operating an experimental unit, this ideal condition cannot be maintained because coking tests are made intermittently, and flue temperatures drop between tests because of excessive radiation from the oven floor. Therefore, in order to duplicate commercial operating results, it is necessary to raise the flue temperature to a minimum of 2200°F, each time before dropping a coal charge into the oven. This temperature is obtained by placing insulation over the oven floor the day before a test is to be made and removing the insulation just before the coal is charged.

When a test run is started, coal is poured through the charge hole and leveled off to the desired depth on the oven floor. The oven door is closed and mudded, the charge hole cover is sealed in place, and the gas exhauster is started. The coal has an initial chilling effect when charged into the oven, but quickly forms a plastic layer which is an excellent insulator and prevents the flue temperature from falling below 2000°F. Gas evolution starts immediately, and a constant pressure of 0.02 inch of water is maintained in the oven chamber by regulation of a by-pass valve around the exhauster. Likewise, a positive pressure of 0.5 inch of water is maintained at the meter outlet to prevent any leakage of air into the gas stream at any point in the scrubbing system.

The rate of evolution and the B.t.u. value of the gas, both of which are at a maximum at the start of a run, gradually decrease over about 60% of the carbonizing period, or until the plastic zone starts to break through the top of the coal bed. At this point both of these values increase slightly over a short period and then decrease rather rapidly until completion of the test.

In all carbonization tests, coking is continued until gas evolution has dropped to a rate of 10 to 15 cubic feet per hour, and the heating value has decreased to approximately 400 B.t.u. per cubic foot. At this point the oven is opened and the coke is pulled out, dropped on an iron plate, and quenched with water. It is dried, weighed, sized, and sampled for both chemical and physical tests. The tar is drained from the separator, water content is determined, and dry volume is computed and corrected to 60°F. The total volume of purified gas, as metered, is corrected to standard temperature and barometric conditions. By carefully controlling the operating conditions as described, and particularly by maintaining constant temperatures and pressures, consistent carbonizing results can be obtained.

CORRELATION OF EXPERIMENTS WITH COMMERCIAL PRACTICE

To correlate experimental results with expected commercial practice, the coals carbonized at the West Frankfort and Millstadt plants were studied. Coal used at West Frankfort is 7/16 inch × 0 raw carbon from Old Ben No. 8 mine, and that used at Millstadt is 1½ inch × 0 washed screenings from the Midwest Radiant Corporation mine located on the property. The
Table I. Percentage Analysis of Coals and Cokes

<table>
<thead>
<tr>
<th></th>
<th>West Frankfort Coal</th>
<th>Millstadt Coal</th>
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<tbody>
<tr>
<td></td>
<td>Coal</td>
<td>Exptl. oven coke</td>
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<tr>
<td>Moisture</td>
<td>9.1</td>
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<tr>
<td>Volatiles</td>
<td>31.2</td>
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<td>Fixed C</td>
<td>49.1</td>
<td>80.8</td>
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<tr>
<td>Ash</td>
<td>10.6</td>
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<td>Total S</td>
<td>1.37</td>
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<td>Average No. Samples</td>
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Table II. Carbonization Results

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<thead>
<tr>
<th></th>
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<tr>
<td>Coke Oven</td>
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<td>1F</td>
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<td>2060</td>
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<tr>
<td>Maximum</td>
<td>3550</td>
<td>2200</td>
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<td>Hours of burn</td>
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<tr>
<td>Gas</td>
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<td></td>
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<tr>
<td>Yield, cu. ft./lb. dry coal at 500 B.t.u.</td>
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<td>4.20</td>
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<td>Actual B.t.u.</td>
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<td>9.0</td>
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<tr>
<td>Coke</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yield, % of dry coal</td>
<td>72.3</td>
<td>72.6</td>
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<tr>
<td>Breeze, % of total coke</td>
<td>19.5</td>
<td>20.5</td>
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<tr>
<td>Shatter test</td>
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<tr>
<td>Over 2 in.</td>
<td>66.2</td>
<td>67.9</td>
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<tr>
<td>Over 1½ in.</td>
<td>83.4</td>
<td>82.1</td>
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<tr>
<td>Apparent sp. gr.</td>
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<td>1.92</td>
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<td>Porosity</td>
<td>64.7</td>
<td>64.3</td>
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Table II. Carbonization Results

<table>
<thead>
<tr>
<th></th>
<th>West Frankfort</th>
<th>Millstadt</th>
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<td>Experimental</td>
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<tr>
<td>Coke Oven</td>
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<td>4E</td>
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<td>Flue temp., °F</td>
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<td>2020</td>
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<tr>
<td>Minimum</td>
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<td>2200</td>
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<td>Maximum</td>
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<td>2200</td>
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<tr>
<td>Hours of burn</td>
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<td>9</td>
</tr>
<tr>
<td>Gas</td>
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<tr>
<td>Yield, cu. ft./lb. dry coal at 500 B.t.u.</td>
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<td>4.51</td>
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<td>Tar yield, gal./ton dry coal</td>
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<td>14.3</td>
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<td>Coke</td>
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<td></td>
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<tr>
<td>Yield, % of dry coal</td>
<td>66.2</td>
<td>67.1</td>
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<tr>
<td>Breeze, % of total coke</td>
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<td>7.9</td>
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<tr>
<td>Shatter test</td>
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<tr>
<td>Over 2 in.</td>
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<td>47.1</td>
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<td>Over 1½ in.</td>
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<td>1.91</td>
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<td>Porosity</td>
<td>55.7</td>
<td>59.4</td>
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<td>Coke Oven</td>
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<td>Depth of coal charge, in.</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>Flue temp., °F</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>Minimum</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Maximum</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>Hours of burn</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Gas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yield, cu. ft./lb. dry coal at 500 B.t.u.</td>
<td>4.7*</td>
<td></td>
</tr>
<tr>
<td>Actual B.t.u.</td>
<td>560</td>
<td></td>
</tr>
<tr>
<td>Tar yield, gal./ton dry coal</td>
<td>13.5</td>
<td></td>
</tr>
<tr>
<td>Coke</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yield, % of dry coal</td>
<td>70.5*</td>
<td></td>
</tr>
<tr>
<td>Breeze, % of total coke</td>
<td>19.0*</td>
<td></td>
</tr>
<tr>
<td>Shatter test</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Over 2 in.</td>
<td>63.1</td>
<td></td>
</tr>
<tr>
<td>Over 1½ in.</td>
<td>75.5</td>
<td></td>
</tr>
<tr>
<td>Apparent sp. gr.</td>
<td>0.872</td>
<td></td>
</tr>
<tr>
<td>True sp. gr.</td>
<td>1.92</td>
<td></td>
</tr>
<tr>
<td>Porosity</td>
<td>54.7</td>
<td></td>
</tr>
</tbody>
</table>

* Estimated.

small experimental oven is operated with closer measurement of the gas, coke, and by-product yields than are the individual ovens of a commercial plant. However, there is enough information on commercial yields to make fairly reliable comparisons with experimental results.

Analysis of coals used in these two plants are shown in Table I, together with analyses of cokes produced from these coals in both experimental and commercial ovens. Carbonizing results obtained both experimentally and commercially are shown in Table II. There is good agreement in most instances between experimental and commercial results. The discrepancies may be explained by differences in coal samples (for example, weathering and fusain content) and by differences in handling the products. If such differences could be eliminated, still better agreement would doubtless be obtained.

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