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CARBONIZING PROPERTIES OF HENRYETTA BED COAL FROM ATLAS NO. 2 MINE, ~~OKMULGEE,~~ HENRYETTA OKMULGEE COUNTY, OKLAHOMA

(Preliminary Report)

By

Joseph D. Davis and D. A. Reynolds

This report represents the results of work done under a cooperative agreement between the Bureau of Mines, United States Department of the Interior, and the Oklahoma Geological Survey

NORMAN

December, 1941

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CARBONIZING PROPERTIES OF HENRYETTA-BED COAL FROM ATLAS NO. 2 MINE, HENRYETTA, OKMULGEE COUNTY, OKLAHOMA[a]

(Preliminary Report)

By

Joseph D. Davis[b] and D. A. Reynolds[c]

This report gives results of tests by the Bureau of Mines-American Gas Association method[1/] at 500° and 900° C. on Henryetta-bed coal from Atlas No. 2 mine, Henryetta, Okmulgee County, Oklahoma, and also results of blending tests at 900° C. with Lower Hartshorne-bed coal from Great Western mine, Sebastian County, Arkansas, and with Upper Hartshorne-bed coal from Quality mine, Sebastian County, Arkansas. The work on the Henryetta-bed coal is in part fulfillment of a cooperative agreement between the Bureau of Mines and the Oklahoma Geological Survey.

Acknowledgments

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Geology of Henryetta-Bed Coal

By

Robert H. Dott[d]

The Henryetta bed coal is a member of the Senora formation, Des Moines Series of Pennsylvanian rocks. The coal extends from Canadian River, near the town of Calvin, southern Hughes County, to Arkansas River, southwest of Coweta, in southwestern Wagoner County. Coal occurs at approximately the same stratigraphic position to the north of Arkansas River, but exact equivalence has not yet been definitely established. The principal district in which this coal is mined is in the vicinity of the city of Henryetta, Okmulgee County. Southward it becomes too thin to be mined on a commercial scale.

"Mines now being operated in the Henryetta district are located near or within a few miles of the outcrop of the coal and the shafts range in depth from less than a hundred feet to three hundred feet. Those mines that are located nearest the outcrop of the bed are slope mines. The coal that is being worked averages 34 to 36 inches in thickness and for the most part is good clean coal from top to bottom. However, in a few places 2 to 4 inches of impure or bony coal are found in the center of the bed. Just north of Schulter the coal becomes extremely faulty and broken, so much that the coal cannot be economically recovered. This condition is maintained almost to Okmulgee; at Bald Hill and Morris the coal is again regular, clean, and of good quality........ Just west of the mined area, six diamond-drill holes have been put down to test out the occurrence of the coal and they show that the Henryetta bed becomes thicker (39 inches in two of the holes) and is of better quality than that found near the outcrop. These drill holes found the coal at a depth of 580 to 630 feet below the surface. These borings show that the coal in this district is not in any way near exhaustion and that there are thousands of tons of coal yet to be mined............"[2]

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d/ Director, Oklahoma Geological Survey.

TABLE 1. Analyses of Henryetta-bed Coal and Blends with Lower and Upper Hartshorne-bed Coals1/

Coal 68 - 100 percent Henryetta bed, Atlas No. 2 mine, Okmulgee County, Okla.
Coal 68A - 80 percent Henryetta bed and 20 percent Lower Hartshorne bed.
Coal 68B - 70 percent Henryetta bed and 30 percent Lower Hartshorne bed.
Coal 68C - 80 percent Henryetta bed and 20 percent Upper Hartshorne bed.
Coal 68D - 70 percent Henryetta bed and 30 percent Upper Hartshorne bed.
Coal 69 - Lower Hartshorne bed, Great Western mine, Sebastian County, Ark.
Coal 70 - Upper Hartshorne bed, Quality mine, Sebastian County, Ark.

Coal No.	Dry mineral-matter-free, fixed carbon	Condition2/	Proximate, percent				Ultimate, percent					Air drying loss, percent	Heating value, B.t.u. per pound	Softening temperature of ash, °F.
			Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur			
68	62.0	1	6.8	33.9	54.0	5.3	5.6	72.5	1.8	12.7	2.1	2.4	12,970	2,040
		2	—	36.4	57.9	5.7	5.2	77.8	1.9	7.2	2.2	—	13,920	—
		3	—	38.6	61.4	—	5.5	82.5	2.0	7.7	2.3	—	14,760	—
68A	66.3	1	5.6	30.4	58.4	5.6	5.3	75.0	1.8	10.5	1.8	2.2	13,230	2,060
		2	—	32.2	61.9	5.9	4.9	79.4	1.9	6.0	1.9	—	14,010	—
		3	—	34.2	65.8	—	5.2	84.4	2.0	6.4	2.0	—	14,890	—
68B	68.2	1	5.3	28.7	60.0	6.0	5.1	75.3	1.7	10.1	1.8	1.3	13,280	2,020
		2	—	30.3	63.3	6.4	4.8	79.5	1.8	5.6	1.9	—	14,010	—
		3	—	32.4	67.6	—	5.1	84.9	1.9	6.1	2.0	—	14,960	—
68C	66.2	1	5.7	30.5	58.4	5.4	5.4	75.0	1.8	10.7	1.7	1.3	13,280	2,050
		2	—	32.4	61.8	5.8	5.0	79.6	1.9	5.9	1.8	—	14,090	—
		3	—	34.3	65.7	—	5.3	84.5	2.0	6.3	1.9	—	14,950	—
68D	68.3	1	5.2	28.8	60.6	5.4	5.2	76.0	1.8	9.9	1.7	.9	13,420	2,010
		2	—	30.3	64.0	5.7	4.9	80.1	1.9	5.6	1.8	—	14,150	—
		3	—	32.2	67.8	—	5.2	84.9	2.0	6.0	1.9	—	15,000	—
69	81.9	1	1.6	17.1	73.5	7.8	4.2	81.7	1.7	3.4	1.2	1.1	14,020	2,420
		2	—	17.4	74.6	8.0	4.1	83.0	1.7	1.9	1.3	—	14,250	—
		3	—	18.9	81.1	—	4.4	90.2	1.9	2.1	1.4	—	15,480	—
70	80.9	1.	1.6	18.3	74.9	5.2	4.5	83.9	1.8	3.9	.7	.9	14,470	2,470
		2	—	18.5	76.2	5.3	4.4	85.2	1.8	2.6	.7	—	14,700	—
		3	—	19.6	80.4	—	4.7	90.0	1.9	2.6	.8	—	15,510	—

1/ Analyses by H. M. Cooper, chemist, Bureau of Mines
2/ 1, Sample as received; 2, moisture-free; 3, moisture- and ash-free.

TABLE 2. Yields of Carbonization Products, as Carbonized Basis

Coal No.2/	Retort diameter, inches	Carbonizing temperature, °C.	Yields, percent by weight of coal1/							Yields per ton of coal				
			Coke	Gas	Tar	Light Oil	Free ammonia	Liquor	Total	Gas, cubic feet	Tar, gallons	Light oil, gal. In gas	Light oil, gal. Tar to 170°C.	$(NH_4)_2SO_4$ pounds
68	13	500	72.0	5.8	8.6	0.36	0.015	12.0	98.8	2,550	20.2	1.14	1.29	3.3
68	18	900	64.6	15.2	5.7	1.09	.098	11.8	98.5	10,450	11.4	2.99	.87	27.8
68A	18	900	68.6	15.0	4.1	1.06	.130	9.4	98.3	10,750	8.4	2.87	.48	24.5
68B	18	900	70.6	14.8	3.6	.96	.131	8.5	98.6	10,700	7.3	2.61	.35	23.3
68C	18	900	68.6	15.0	4.5	1.08	.166	9.6	99.0	10,650	9.2	2.95	.47	26.9
68D	18	900	70.0	15.1	4.0	1.04	.163	8.8	99.1	11,050	7.9	2.83	.37	26.4

1/ Coke, tar, ammonia and light oil are reported moisture-free; gas is reported as stripped of light oil and saturated with water vapor at 60° F. and under 30 inches of mercury.

TABLE 3. Analysis of Coke, Dry Basis

Coal No.2/	Retort diameter, inches	Carbonizing temperature, °C.	Proximate, percent		Ultimate, percent						Heating value, B.t.u. per pound
			Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur	
68	13	500	10.5	82.4	7.1	3.0	82.8	2.2	3.3	1.6	13,800
68	18	900	1.4	90.0	8.6	.7	87.0	1.7	.2	1.8	13,180
68A	18	900	1.8	89.7	8.5	.7	87.4	1.6	.0	1.8	13,230
68B	18	900	1.9	89.2	8.9	.7	87.2	1.5	.0	1.7	13,190
68C	18	900	1.6	90.6	7.8	.7	88.3	1.6	.0	1.6	13,300
68D	18	900	1.5	90.8	7.7	.6	88.5	1.5	.2	1.5	13,290

2/ See table 1 for explanation of sample numbers.

Chemical Composition of Coals and Blends

Table 1 shows the chemical composition and heating value of coals and blends studied in this investigation and also the softening temperatures of the ashes. The rank of Henryetta coal (No. 68) is high-volatile B; both of the Hartshorne coals (Nos. 69 and 70) are low-volatile. In general, high-volatile B coals are not used for making blast-furnace coke when coals of higher rank are available at reasonable cost, but it is true in a few instances that such coals are being used successfully. The American Society for Testing Materials classifies coals for coke-making[3/] upon the basis of the ratio of fixed carbon to volatile matter and states that this ratio for coals suitable for the purpose ranges from 1.4 to 5.0; that for Henryetta coal is 1.6. The sulfur content of Henryetta coal (2.1%) is higher than that of coals ordinarily used for making metallurgical coke, but it is not believed to be high enough to render the coal unfit for that purpose; it is reduced somewhat by blending with the Hartshorne coals. The softening temperature of the ash is low, and it is also low in the blends with the Hartshorne coals, the ashes of which soften at higher temperatures. It is not possible to predict the softening temperatures of ashes of blends from those of the constituent coals; however, softening temperatures of ashes from the cokes do not differ appreciably from those of the coals.[4/] It is believed that coke from either Henryetta coal or blends of Henryetta and Hartshorne coals would not prove suitable for water-gas generator fuel because of the low softening temperature of the ash and that it would clinker considerably if used for industrial or domestic heating. The American Society for Testing Materials[5/] states that the softening temperature of ash in coke for water-gas generator fuel should be above 2,300° F., and that the ash in cokes for domestic and industrial heating should not soften below 2,200° F. The softening temperature of the ash is not important in blast-furnace coke.

Yields of Carbonization Products

Table 2 gives the yields of carbonization products obtained from Henryetta coal at 500° and 900° C. and from the blends at 900° C. The yields of these products from the Henryetta coal are satisfactory, considering its rank; that of ammonium sulfate is exceptionally high (27.8 pounds per ton) and it is not reduced greatly by blending with the Hartshorne coals. The yield of liquor is high, and this may be the cause of the high yield of ammonium sulfate; the steam formed in the retort tends to prevent decomposition of ammonia. The yield of gas by volume is normal for a coal of this rank, and it actually is increased by blending with the low-volatile Hartshorne coals. Usually reduction of the volatile content by blending decreases the yields of both gas and tar. The yield of light oil is high; that for the Henryetta coal and for the blends, including light oil in the tar, is more than 3 gallons per ton, except for blend B.

Analysis of the Coke

Table 3 gives analyses of the cokes upon the dry basis. The main constituents of interest in this table are the ash and sulfur. The ash content is within the range covered in commercial cokes made in the United States, but the sulfur content is somewhat high; the American Society for Testing Materials5/ specifies that coke for blast furnaces shall not contain more than 1.3 percent of sulfur. Blast-furnace operators usually are liberal in their tolerance of ash, but it is true that ash in the coke reduces the amount of carbon available for smelting the ore and that limestone is required to flux ash.

Physical Properties of the Coke

Table 4 shows the physical properties of the cokes. This table is important because one of the main objectives of the work was to determine whether the Henryetta and similar Oklahoma coals would produce coke strong enough for blast-furnace use in connection with National Defense. This coal does not produce coke as strong as do high-volatile Appalachian coals ordinarily used for making blast-furnace coke, but it is believed that it can be used for that purpose. At least the coke

TABLE 4. Physical Properties of Coke

Coal No.[2]	Retort diameter, inches	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon 2-inch screen	Shatter test, $1\frac{1}{2}$-inch screen	Shatter test, 1-inch screen	Shatter test, $\frac{1}{4}$-inch screen	Tumbler test, cumulative percent upon 2-inch screen	Tumbler test, $1\frac{1}{2}$-inch screen	Tumbler test, 1-inch screen	Tumbler test, $\frac{1}{4}$-inch screen
68	13	500	1.49	0.70	53.0	73.4	83.7	89.4	94.3	11.7	33.3	45.2	49.9
68	18	900	1.89	.78	58.7	18.3	46.6	82.3	97.9	.0	2.4	36.3	76.3
68A	18	900	1.92	.81	57.8	43.6	73.5	92.0	98.2	1.9	24.2	58.7	74.8
68B	18	900	1.92	.83	56.8	51.1	77.4	94.2	98.5	4.7	29.1	62.9	75.2
68C	18	900	1.90	.83	56.3	41.1	76.7	90.0	98.3	1.4	29.0	62.5	77.0
68D	18	900	1.91	.82	57.1	44.4	75.6	93.7	98.5	2.9	25.9	61.9	77.7

TABLE 5. Physical and Chemical Properties of Gas

Coal No.[2]	Retort diameter, inches	Carbonizing temperature, °C.	Specific gravity	Gross heating value[1]: B.t.u. per cubic foot, Determined	Gross heating value: B.t.u. per cubic foot, Calculated	Gross heating value: B.t.u. per pound of coal	H_2S, grains per 100 cubic feet	Composition, dry, percent by volume: CO_2	Illuminants	O_2	H_2	CO	CH_4	C_2H_6	N_2
68	13	500	0.603	835	826	1,060	3,360	7.2	3.0	0.5	22.8	4.8	49.1	11.7	0.9
68	18	900	.385	567	560	2,960	670	2.9	4.1	.4	53.1	9.0	28.3	1.0	1.2
68A	18	900	.370	542	539	2,910	480	3.0	3.8	.3	55.6	8.4	27.5	.1	1.3
68B	18	900	.367	515	521	2,760	490	3.2	3.3	.3	57.6	8.1	26.2	.0	1.3
68C	18	900	.373	545	544	2,900	470	2.8	3.7	.3	55.4	8.1	28.6	.0	1.1
68D	18	900	.363	533	532	2,940	480	3.0	3.5	.3	56.5	7.9	27.1	.2	1.5

1/ Stripped of light oil and saturated with water vapor at 60° F. and under 30 inches of mercury.
2/ See table 1 for explanation of sample numbers.

TABLE 6. Analysis of Dry Tar

Coal No.1/	Retort diameter, inches	Carbonizing temperature, °C.	Specific gravity, 15.6°C./15.6°C.	Heating value, B.t.u. per pound	Percent by weight: Ultimate analysis: Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur	Free-carbon	Anthracene salts	Naphthalene salts	Boiling range, °C., percent by volume: 0-170	170-235	235-270	270-350	Residue
68	13	500	1.02	16,540	8.9	83.0	0.8	6.8	0.5	0.57	0.00	0.00	6.4	28.3	10.4	22.3	32.6
68	18	900	1.15	16,180	6.1	88.1	1.4	3.6	.8	4.87	1.60	3.01	7.6	21.5	5.0	15.6	50.3
68A	18	900	1.15	16,150	5.9	88.8	1.3	3.3	.7	3.85	2.39	4.01	5.7	21.0	5.7	16.8	50.8
68B	18	900	1.16	16,170	5.8	89.1	1.3	3.1	.7	3.75	2.67	4.54	4.8	20.7	5.6	17.0	51.9
68C	18	900	1.16	16,140	5.8	88.7	1.4	3.3	.8	4.71	2.02	3.59	5.1	20.4	5.7	15.9	52.9
68D	18	900	1.16	16,220	5.7	89.3	1.3	3.0	.7	4.13	2.74	4.75	4.7	19.7	5.6	15.7	54.3

TABLE 7. Analysis of Tar Distillate and Light Oil

Coal No.1/	Retort diameter, inches	Carbonizing temperature, °C.	Distillate, percent by volume of dry tar: Acids	Bases	Neutral oils	Neutral tar oil percent by volume: Olefins	Aromatics	Paraffins and naphthenes	Refined light oil from gas, percent by volume: Benzene	Toluene	Paraffins	Solvent naphtha	Crude light oil from gas: Olefins, percent by volume	Naphthalene percent by weight
68	13	500	28.2	1.7	37.6	10.3	55.1	34.6	10.4	12.0	51.6	26.0	33.3	0.00
68	18	900	9.8	2.4	32.9	10.8	88.1	1.1	72.7	20.3	1.6	5.4	10.4	.06
68A	18	900	8.5	2.6	31.7	13.0	86.6	.4	71.8	21.5	1.0	5.7	9.8	.00
68B	18	900	7.9	2.6	30.4	12.6	87.0	.4	71.3	22.2	.8	5.7	9.9	.08
68C	18	900	8.3	2.5	30.6	10.8	88.5	.7	71.7	22.0	.9	5.4	10.0	.10
68D	18	900	6.6	2.5	29.0	11.4	88.2	.4	73.2	21.3	.5	5.0	8.7	.13

1/ See table 1 for explanation of sample number.

possesses somewhat better physical properties than does that from coal No. 19[6] of the BM-AGA survey of carbonizing properties of American coals which is known to have been used successfully in a blast furnace. The table shows that an enormous improvement in strength of coke, as measured by shatter and tumbler tests, is realized by blending with 20 percent of low-volatile Hartshorne coal, which is available and can be used for that purpose if desired. Use of 30 percent of the low-volatile coals (blends B and D) does not exhibit clear-cut improvement over results realized in the 20 percent blends (A and C), and it is doubtful if 30 percent blends would be satisfactory because, as will be shown later, these low-volatile coals are strongly expanding.

Physical and Chemical Properties of the Gas

Table 5 shows the physical and chemical properties of the gas. In view of the abundance of natural gas in Oklahoma, these results may appear to be of minor interest at present; nevertheless, this gas could and should be utilized. It is an established principle in by-product coking that every product must be made to yield income, however small, to help reduce the cost of the coke. Probably this gas would be used for underfiring the coke ovens or for fuel in the manufacturing plant.

The Henryetta coal is not a good gas coal; such materials should yield more than 3,000 B.t.u. in gas per pound of coal carbonized. Furthermore, the sulfur content of the gas is high, even for the blends. It should not be higher than 250 grains per 100 cubic feet and preferably less. The yield of gas upon the B.t.u.-per-pound basis is not reduced by blending; it has been shown that it actually is increased upon the volume basis.

Properties of Tar

Tables 6 and 7 show the properties of the tars, and they do not differ appreciably from those obtained from other coals of similar rank, except that the tar-acid content of the 500° C. tar is high. The yield of this tar was 20.2 gallons per ton of coal; accordingly, there would be available 20.2 x 0.282 = 5.7 gallons of tar acids per ton if carbonized at this low temperature. Low-temperature carbonization processes yield high percentages of tar acids, which can often be used to advantage for making plastics.

Expanding Properties

Henryetta coal contracted 3.3 percent in the Bureau of Mines sole-heated test oven at a charge density of 55.5 pounds per cubic foot, whereas the Upper Hartshorne expanded 38.8 percent and the Lower Hartshorne 33.9 per cent. High-volatile coals virtually always contract, whereas low-volatile coals expand. Under these conditions of test[7] a coal contracting 14.0 percent is considered moderately contracting and one that expands 10 percent is considered moderately expanding. The blends tested in this work therefore consisted of mixtures of a weakly-contracting coal with strongly expanding coals. It is not known that the expansion of a blend equals the algebraic sum of the expansion of coals used, but large proportions of strongly expanding coals in blends are known to have damaged coke-oven walls. All of the blends tested in this work deformed the steel retorts moderately, and this is a characteristic of expanding blends. The deformation was least in blends containing Lower Hartshorne coal. Because of the expanding properties of the Hartshorne coals it may not be advisable to use even as much as 20 percent in blends, particularly if the ovens used are narrow and flue temperatures are high. However, smaller percentages should increase the strength of the coke appreciably.