

PILOT PLANT STUDIES ON THE PRODUCTION OF METALLURGICAL COKE. (*)

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Coke is an essential raw material for the manufacture of iron by the conventional blast furnace technique.

Due to the paucity of reserves of high grade coking coals suitable for the production of metallurgical coke a rational use of these types of coals and utilization of increasing proportions of substandard coals for coking are receiving serious consideration in most countries in the world.

Until the fundamental principles underlying coke formation from carbonisation of coals are clearly understood investigation on the selection of coals for coke-making will largely remain a method of trial and error. But exploratory experiments on the production of coke are difficult to be carried out in commercial plants for various reasons. The question of cost and interference with normal plant operation are two of the greatest handicaps. In this connection, the famous saying of Backeland may aptly be quoted 'Commit your blunder on a small scale and make your profits on a larger scale'.

Accordingly efforts have been made from early days of the carbonising industry to obtain useful information of a preliminary nature from tests carried out in the laboratory or small scale pilot plants. Apart from qualitative and quantitative studies of the products obtainable from coals from different sources and their blends treated under different carbonising conditions, these pilot oven tests afford information of vital importance to the detailed design of commercial plants of modern days with all its intricacies.

Even if large scale tests in commercial ovens be otherwise possible it is difficult to carry out the investigations strictly under any desired test conditions because of the great bulk of the sample coals required to be handled and unavoidable snag in operating industrial plants.

A general survey of small-scale experimental ovens both gas-fired and electrically heated has already been made in an earlier paper. A short description of a few more are presented here.

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Reference was made in the earlier paper of a multi-oven battery of pilot plant being installed in the Central Fuel Research Institute. This has since been put into commission. A brief description of this plant and its working is also given in this paper.

Data obtained from comparative tests carried out in pilot and commercial ovens are presented showing the correlation between the two.

DESCRIPTION OF PILOT OVENS

United States Steel Corporation:

For several years, the Applied Research Laboratory of the United States Steel Corporation has successfully made use of a 30 lbs. electrically heated test oven wherein straight coals as well as coal blends are carbonised and the physical strength of the cokes produced is determined.

The effects of different carbonising conditions on the strength of the coke are also studied.

Cerchar-France :

In France, Centre d' etudes et Recherches des Charbonnage has devised a 20 kg. test oven. The electrically heated oven is constructed to accommodate a retort of 20 kg. capacity wherein coal is carbonised at a temperature range of 950 to 1300°C. The retort is provided with thermocouples at different positions inside the retort to measure the temperature of the coal charge, during carbonisation. The retort is of cylindrical shape, lined inside with refractory material and has a diameter of 420 mm. and a length of 300 mm. between the two ends. The bulk density of the coal charge is varied 800 kg/m³ to 1000 kg/m³.

British Coke Research Association - U.K.

The British Coke Research Association has reported the use of 15 lbs. gas-fired pilot oven for carrying out carbonisation tests of coal and coal blends from various sources. Three ovens have been designed and constructed, two being side-heated and the third sole-heated. The oven widths are 3 inch and 4½ inch capable of accommodating 10-15 lbs. coal charges. The sole heated oven is devised to measure the expansion and contraction property of coal. A movable top plate registers on a paper the degree of expansion and contraction. The other two ovens were designed to carbonise coal by side-heating and were effectively miniature coke ovens. Effects of charging condition and bulk density of the charge on the physical strength of coke are studied.

Resources Research Institute- Japan :

In the bulletin of the Resources Research Institute, Japan the use of an electrically heated oven for carrying out small-scale carbonisation tests is described. 1.5 kg. coal is put in an iron retort and charged in an oven at a temp. of 700°C. The temperature of the oven is thereafter raised to 1000°C. Description is also given of gas-fired test ovens having capacities ranging from 250 kg. to 400 kg. The width of the carbonising chambers vary from 400 mm. to 450 mm. In these ovens, as well coal blends from different sources are carbonised and their strength tested.

Stein Kohlenberghauverein- Germany:

In the research laboratory of "Steinkohlenberbauverein", Essen in Germany, an experimental gas-fired oven having dimensions of 450 mm. width, 100 mm. length and 700 mm. height has recently been set up. Coal blends of 10% water content are carbonised at temperatures varying between 900°C and 1250°C. and the physical properties of the coke produced are compared. Tests are also carried out at oven temperature comparable with commercial ovens. Comparative studies are made of the physical strength and abrasability of the coke produced in this pilot plant and in commercial ovens. Effects of varying bulk densities, particle size and water content of the coal charge on the physical strength of coke are also studied.

Pilot Plant at C.F.R.I. - India :

This pilot plant was installed in the Central Fuel Research Institute in the year 1957. It consists of three ovens with regenerative system of heating, built of silica bricks having widths of 14 in., 16 in. and 18 in. having capacities of 980 kg., 1100 kg. and 1180 kg. respectively. Both in its construction and working it simulates modern industrial plants almost in all respects.

There is arrangement for selective crushing of coal in a specially designed impact type hammer mill with provision for recycling of the oversize coal over vibrating screens so as to make the entire material pass through any desired mesh. There are over-head bunkers for separate storage of coals of different types and a conical mixer is provided for blending predetermined proportions of coals. There is an electrically driven ram car for pushing the coal charge and the coke is manually quenched on a sloping wharf lined with special bricks.

The gaseous products of carbonization pass through cast iron ascension pipes and foul gas main to primary coolers and then to an electrostatic detarrer. The gas is thereafter sucked by an

exhauster which on the other hand delivers it to a final cooler and then through an ammonia scrubber. Finally the gas is stored in a 150 m³ capacity gas holder wherefrom it is recycled for use for underfiring. The condensed tar and liquor from the gas mains, coolers and electric detarrer are collected in tar catch tanks.

Majority of the test ovens including those described in the earlier paper which are in use in different countries are principally meant for a study of the quality of coke alone and are not generally provided with facilities for studies of the by-products. The plant newly installed in the C.F.R.I. is unique in that, it is a complete battery of coke ovens and affords detailed assessment of all the products obtained from carbonisation of coals.

The plant is completely instrumented for continuous recording of temperature, pressure, drafts etc. and is provided with automatic safety blender for gas.

Repeatability of Test Results.

The repeatability of results in respect of quality of coke, the main product, obtained from duplicate tests carried out in the multi-oven pilot plant at the C.F.R.I. is shown in Table 1.

Table 1 - Repeatability of Test Results in Pilot Plant.

Properties of Coke	Test Nos.	
	I	II
Screen Analysis		
% cumulative on		
4"	48.7	45.7
3"	74.7	70.9
2"	91.7	90.4
1"	96.4	93.7
$\frac{1}{2}$ "	97.4	95.7
Yield(dry)%	75.4	75.4

Table 1(Contd.)

	Test Nos.	
	I	II
<u>Hardness and Strength Test</u>		
Shatter Index	89.2	88.3
% on 1½"		
% on ½"	98.1	97.5
Micum Index		
% on 40 mm.	75.1	77.0
% through 10MM.	11.1	10.0
Stability Factor		
on 1"	57.7	56.8
% Porosity	42.2	42.8
Bulk density lb/cft.	30.6	30.7
<u>Proximate Analysis (dry) %</u>		
Ash	21.2	21.4
Volatile Matter	1.6	1.2
Fixed Carbon	77.2	77.4

The values for both the chemical and physical properties of the cokes obtained from the two tests are observed to be closely concordant. Comparison of Test Results from Pilot and Commercial Ovens :

The exact carbonising conditions of different experimental ovens have to be initially ascertained by performing preliminary scaling tests so that the results of experiments carried out in these ovens are comparable with those from commercial ovens. In certain cases factors may have to be established correlating the results from experimental and commercial oven tests.

The results of a pair of comparative tests carried out in the 16 in. wide oven of the pilot plant of the Central Fuel Research Institute and in a commercial oven of 16 in. width are discussed below.

Table 2 shows the fairly identical nature of the carbonising conditions maintained during tests in the two types of ovens.

Table 2 - Carbonising Conditions*

	C.F.R.I. Pilot Plant	Commercial Plant (Mean width 16")	
1. Flue Temperature (°C)	1270	1270	
2. Coke Mass Temperature (°C)	1050	1040	
3. Gross Coking Time (Hrs.Mnts)	16.20	16.00	
4. Moisture in Coal Charge (%)	8.0	8.2	
5. Grain Size of Coal	about 90% thru' 3 mm	about 90% thru' 3 mm	
6. Gas Main Pressure (mm. W.G.)	3.0	3.5	
7. Analysis of Waste Gas (% vol/vol)			
	CO ₂	8.3	9.4
	O ₂	3.3	3.1

* The figures represent average values for a test period.

Table 3 compares the properties of coke, tar and gas produced from the pilot plant and commercial oven by carbonising the same kind of coal charges.

Table 3 - Comparison of Results from Tests in Pilot and Commercial Ovens.

(i) Properties of Coke

	OVEN	USED
	*Pilot	Commercial
<u>Hardness and strength Test</u>		
Shatter Index on $1\frac{1}{2}$ " %	90.2	92.6
=do- on $\frac{1}{2}$ in.%	97.1	97.6
Micum Index on 40 mm.%	75.0	76.8
-do- thr.10 mm.%	13.1	9.8
Stability Factor on 1 in.%	49.8	50.0

• (Refer to the coke made in the 16" wide oven)

(ii) Properties of Tar

Specific Gravity	1.16	1.18
Ash free carbon %	7.7	9.3

Fractional Distillation of Tar oil % w/w on dry tar.

Upto 170°C	2.0	0.4
170°C - 230°C	13.4	7.1
230°C - 270°C	9.6	11.1
270°C - 360°C	26.8	20.5
Crude Tar acid	6.2	2.8
Crude Naphthalene	3.4	7.2

(iii) Properties of Gas vol.%

CO ₂	3.7	3.3
C _n H _m	3.6	2.6
O ₂	0.5	0.3
CO	7.9	8.5
H ₂	48.6	49.2
CH ₄	31.5	33.0
N ₂	3.2	3.1
Specific Gravity	0.44	0.40
Calorific Value; (B.thu/cft.)	500	510

The following salient features may be noted from the above.

∠bulk
Coke: The physical properties of the coke from the commercial oven are observed to be slightly better than those produced in the pilot plant. This is particularly true of the minus 10 mm. Micum Index and is largely due to the greater density of the coal charged in a commercial oven as compared with pilot plants of smaller height. There is also a trend of decrease in the percentage porosity of the coke possibly due to the same reason.

By-products: Owing to the fact that the pilot plant under consideration has three ovens of different width it is not possible to compare strictly the products other than coke with those from a commercial battery having all its ovens of the same width. Bearing this limitation in mind, it may still be seen that the properties of the gas produced in the pilot and commercial oven are about the same.

The tar from the pilot plant has invariably a lower specific gravity, a higher tar-acid content and a lower naphthalene and lower free carbon contents. This is possibly a result of less cracking of the tar in the small pilot plant.

Study of Influence of Factors on the Coke Quality: *

Since the principle objective of tests in a pilot plant is to investigate the possibility of utilizing maximum proportion of poorer coking coals, the carbonising conditions under which best results can be obtained in actual commercial practice have to be found out.

It thus becomes necessary to investigate the influence of the different factors such as :-

- (a) the influence of the fineness of the coals and the moisture content of the charge.
- (b) the carbonising temperature
- (c) the oven width or the rate of carbonisation,
- (d) the bulk density of the charge,
- (e) the proportion of the inferior coking coals in the coal charge.

Such studies are being made in the pilot ovens which are in operation at the C.F.R.I and in Jamshedpur. The results obtained have proved to be of invaluable help to the planning of the new coke plants which have been installed in the country in recent years.

Some of the more important findings of researches carried out so far in these pilot plants have already been implemented in the commercial plants with the ulterior object of conserving metallurgical coal.

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