Mr. H. M. Woodrooffe

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MINES BRANCH INVESTIGATION REPORT IR 62-99

RECOVERY OF CARBON FROM "DOLOCHAR" (PROJECT MP-IM-6202)

by

R. A. WYMAN

MINERAL PROCESSING DIVISION

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SUMMARY OF RESULTS

Up to 94% of the carbon in "dolochar", the non-magnetic residue of the direct reduction kiln in operation at the Steel Company of Canada plant at Hamilton, Ont., may be recovered by a system of partial hydration and screening, at grades of up to 86% C. Higher grades are unlikely due to the concentration of ash in the char fragments.

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*Head, Industrial Minerals Milling Section, Mineral Processing Division, Mines Branch, Department of Mines & Technical Surveys, Ottawa, Canada.

INTRODUCTION

Following discussions and correspondence, early in 1962, with members of the Research & Development Department of the Steel Company of Canada, Limited, Hamilton, Ontario, the Mines Branch agreed to investigate the prospects for recovering carbon from the non-magnetic residue of a direct reduction kiln operating at the Steel Company plant.

The kiln is charged with a mixture composed of 40% anthracite coal, 6% dolomite, and the balance sized iron ore pellets. After a kiln residence time of 5 hr the iron has been reduced to magnetite, and the unconsumed coal has largely become "char" (coke or charcoal); the dolomite has become calcined and has absorbed most of the sulphur contained in the original charge. The material discharged from the kiln is run over magnets, screened on 1/4 in. and the -1/4 in. passed through magnetic separators. The remaining non-magnetics are called "dolochar", of which the major constituent is carbon. This carbon has an approximate value of $8.00 per ton. The Steel Company would like to recover the carbon in the form of a 90% C product and discard the dolomite and ash, preferably by a dry process.

SAMPLE

To keep air slaking of the dolomite to a minimum a sample was sent to the Mines Branch in February, 1962 in two sealed drums. Each contained approximately 100 lb. A typical analysis of dolochar as provided by the Steel Company was:

Total Fe	1.30%
S	1.11%
C	70.18%
CaO	8.35%
MgO	5.64%
	86.58%
Ash (assumed)	13.42%
	100.00%

One of the drums was opened and separated into screen fractions with the following result:

+6 mesh	42.2%
-6+10 mesh	35.5%
-10+20 mesh	15.1%
-20+35 mesh	3.3%
-35+65 mesh	0.9%
-65 mesh	3.0%
	100.0%

TEST WORK

The original conception of this problem was separation by air table, or possibly by air separator. Four pilot tests were run on the Whippet air table, one with each of the following screen fractions of the feed viz: +6 mesh, -6+10 mesh, -10+20 mesh, and -20+35 mesh. Some tendency for carbon and dolomite to separate was observed in each screen size, but with the best separation on the +6 mesh. Additional tests were, therefore, run on the larger size air table (Kipp Kelly), with +6 mesh material and -6+10 mesh material.

The best product from the Kipp Kelly, representing 28% of the +6 mesh feed, still had observable particles of dolomite in it. The poorest product, representing 2.4% of the feed, was at least half dolomite. Results with the -6+10 mesh were not as good as for the +6 mesh. A test was run on the air jig with +6 mesh feed with poorer results than the air table. The visual evidence was enough to rule out this approach and no analyses were obtained.

Trials were then begun on the effect of hydration on the dolomite. Grains of dolomite were separated from the carbon by hand. Water alone had little immediate effect on these grains but when they were heated, a few drops of water caused them to break up into smaller pieces at once.

Several trials were made of heating +10 mesh dolochar in a muffle furnace, dumping it into a pan, and spraying it with water. Heating to 300°C and spraying with a mist had no effect. Spraying with a coarse droplet spray also failed to cause breakdown of the dolomite. Heating to 500°C and spraying also failed. Some selected dolomite grains were heated in the same furnace to 300°C and a few drops of water were added. Good slaking was achieved. It was concluded that spraying the charge tended to cool it down before water droplets had reached more than a few of the dolomite grains.

In the next test +10 mesh feed was heated to 300°C and a fine stream of water from a wash bottle was used. This caused a much better breakdown of the dolomite and allowed a considerable removal by screening on 28 mesh. A repeat of this test with spraying by multiple jet spray caused excessive dampening, and the dolomite, though slaked, was too sticky to screen out.

Attempts to improve the technique by spraying the heated dolochar as it was discharged from a vibrating feeder, spraving on a vibrating screen, and spraying in a rotating pan were only partially successful.

Attention was next turned to a reversal of the system. A number of trials were made of dampening the dolochar, heating to 300°C then screening. An immediate improvement was observed. On withdrawal from the furnace, the dolomite was seen to have cracked into many small pieces. Screening on 28 mesh removed a considerable amount, but much of the dolomite, though smaller in size than the original pieces, was still larger than 28 mesh. Attempts were made to further reduce the dolomite particle size by placing objects on the vibrating screen. Washers, small rubber balls ($\frac{1}{2}$ in. diameter), large rubber balls (1 in. diameter), and $\frac{1}{2}$ in. burundum pellet were all tried. The best results were obtained with burundum pellets, although some carbon was also reduced to -28 mesh and passed through the screen.

Several attempts were made to use infra-red heating of the dampened dolochar without further improvement in results. Allowing steam to rise through a screen on which dolochar had been placed caused hydration but left the dolomite too sticky to pass through the screen. Agitating the material on the screen during the steaming process improved the method but did not result in thorough elimination of the dolomite. The pieces reduced in size but not all to -28 mesh. Allowing dampened dolochar to stand in air overnight resulted in the same tvpe of cracking and reduction to smaller size in the dolomite.

Of all the methods tried, that of heating the damp dolochar and then screening, with burundum pellets on the screen, proved the most promising. A system was set up to make trials on a continuous basis. The dolochar was dampened by spraying as it cascaded in a cement mixer. The damp dolochar was fed through a small kiln composed of a sloped stainless steel tube. Movement through this tube was affected by vibration. Passage time was about 1 minute, the hot zone was at about 400°C. The hot dolochar discharged into a Sweco screen on which a 1 pound charge of $\frac{1}{2}$ in. burundum cylinders had been placed. The screen cloth was 28 mesh.

For the first trial of this system +20 mesh dolochar was used as feed. The Sweco screen oversize was rescreened on 20 mesh. Results were as follows:

TABLE 1

Results of 1st Hydration Run

Fraction	Wt %	Free C%	Acid Sol. %	Ash %
+20 m	92.3	81.0	5.3	13.7
-20+28 m	5.0	69.6	19.1	11.3
-28 m	2.7	32.0	54.1	13.9
	100.0	79.0	7.4	13.6

It was apparent that there had been an insufficient removal of dolomite and ash despite the concentration of acid soluble in the -28 mesh. Accordingly the +28 from the above run was repassed through the systems. The results were as follows:

TABLE 2

Rerun of the +28 from 1st Step

Fraction	Wt %	Wt % of Orig. feed	Free C%	Acid Sol. %	Ash %
+20 m	90.0	87.6	85.9	1.7	12.4
-20+28 m	4.5	4.4	69.0	19.2	11.8
-28 m	5.5	5.3	24.3	69.3	6.4
	100.0	97.3	81.7	6.2	12.1

The +20 mesh in the above table represented a recovery of 95% of the carbon in +20 mesh feed, or a recovery of 94% of the carbon in the dolochar. Further trials with +20 mesh starting feed gave almost identical results.

A test was next arranged for dolochar as received. The feed mixture was made quite damp in this test. As before the kiln discharge was screened on 28 mesh, but in addition the

+28 was screened on 14 mesh. Results were as follows:

TABLE 3

Test with Dolochar as Received

Fraction	Wt %	Free C%	Acid Sol. %	Ash %
+14 m	76.4	80.6	3.4	16.0
-14+28 m	13.1	73.4	15.9	10.7
-28 m	10.5	32.3	53.6	14.1
	100.0	74.6	10.3	15.1

Recovery of carbon in the +28 mesh fractions was 95.5%. Neither the +14 nor the -14+28 mesh was, however, considered sufficiently improved in carbon content. Each of these fractions was therefore separately rerun through the system. Results are given below:

TABLE 4

Rerun of Table 3 Products

Fraction Feed	Fraction Product	Wt %	Wt % of Orig. Feed	Free C%	Acid Sol. %	Ash %
+14 m	+28 m	98.7	75.4	83.7	0.9	15.4
	-28 m	1.3	1.0	6.0	90.2	3.8
		100.0	76.4	82.6	2.1	15.3
-14 +28 m	+28 m	90.0	11.8	77.0	13.4	9.6
	-28 m	10.0	1.3	35.7	53.6	10.7
		100.0	13.1	72.8	17.5	9.7

The combined +28 mesh from the above contained 94.4% of the carbon in the dolochar at a grade of 82.9% C. Further trials of the same type failed to improve upon this result.

One further approach was tried, more as an experiment than as a practical solution. The idea in this case was to see if the dolomite would hydrate, and be ground to fines by the tumbling of the charge in the cement mixer.

A batch of the dolochar, as received, was placed in the cement mixer and thoroughly dampened. The mixer was allowed to rotate for 3 hours after which the charge was dumped and screened on 28 mesh. The +28 was returned to the mixer, redampened and tumbled for a second period of 2 hours, then re-screened on 28 mesh. Two further cycles of dumping, tumbling and screening were also completed. The charge was to all intents and purposes dry at the end of each tumbling period. The results of this trial are given below.

TABLE 5

Hydration and Tumbling

Cycle	Fraction	Wt %	Free C%	Acid Sol. %	Ash %
1	-28 m	4.5	49.7	36.8	13.5
2	-28 m	2.7	38.3	48.7	13.0
3	-28 m	1.5	35.7	51.1	13.2
	(+28 m	91.3	76.0	9.1	14.9)*
4	-28 m	0.5	39.9	48.5	11.6
	+28 m	90.8	77.6	7.9	14.5
		100.0	74.5	11.1	14.5

* +28 mesh after the 3rd treatment.

The remaining +28 mesh in the above test contained 94.8% of the carbon at a grade of 77.6% C.

DISCUSSION

Attempts to beneficiate dolochar by dry gravity methods were not at all promising. A method of partial dry hydration and screening succeeded in concentrating the bulk of the carbon without decreasing the ash content to any extent. The best results were obtained by discarding -20 mesh fines contained in the dolochar sample then hydrating the +20 mesh material and rescreening.

It was noted that in all cases ash tended to follow the carbon although most of the dolomite could be discarded in the fines. It was also noted that the ash content, 12-16%, tended to preclude the possibility of a 90% carbon product. The fact that the ash tended to follow the carbon suggested that it was not released in particles of the size forming the product, +28 mesh. It was thought the anthracite coal used to provide carbon in the kiln feed might have 6-10% ash, therefore ash must be increasing in the char type of carbon being discharged from the kiln. To test this, small samples of bright coal and dull char were carefully sorted from the sample and analysed separately. The results of this test amply confirmed the conjecture as indicated below.

TABLE 6

Bright vs Dull Dolochar

Material	Free C%	Ash %
Bright	90.9	6.6
Dull	31.1	64.5

On this basis it was considered that the test results had probably produced grades which were nearly as high as could be expected with this material.

CONCLUSIONS

(1) Dry gravity methods are unlikely to succeed in beneficiating dolochar.

(2) Partial hydration followed by screening will recover up to 94% of the carbon content of dolochar at grades of up to 86% C in what is essentially a dry process.

(3) Grades of much above 86% are unlikely due to the high ash content of the "char" particles in dolochar.

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