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IR 5433

CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 74-33

A MINERALOGICAL INVESTIGATION OF SAMPLES FROM THE CLEAR HILLS OOLITIC IRON DEPOSIT, PEACE RIVER, ALBERTA

by

W. PETRUK, D.C. HARRIS AND R.G. PINARD

MINERAL SCIENCES DIVISION

COPY NO. 31

JULY 30, 1974

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SUMMARY

A sample of iron ore from the Clear Hills deposit in Alberta was studied to determine its mineralogy and the mineralogical characteristics that could affect mineral beneficiation. The sample contains about 16.5 wt.% adsorbed water and when dried at 100° for 3 hours has 37 wt.% Fe and 1.5 wt.% P_2O_5. It consists of about 60 wt.% oolites, 20 to 32 wt.% amorphous iron-bearing earthy matrix, 6 wt.% siderite, 4 wt.% quartz, and 1 to 2 wt.% phosphate. The oolites have a variable mineralogical composition, but an average oolite consists of 54 wt.% goethite, 42 wt.% amorphous iron-bearing gangue, and 4 wt.% quartz and contains about 43 wt.% Fe. Most of the goethite in the oolites is present as very narrow layers thus it is considered that the only practical method of recovering the iron is by concentrating the oolites and siderite. "Quantimet" image analysis show that for this sample 77% of the iron is present as oolites and siderite and if full recovery were achieved, the grade of the resulting concentrate would be 43 wt.% Fe. Size distribution data show that oolite liberation would increase rapidly with grinding to 93% (71% of the iron) at -65 mesh, but 100% liberation would be achieved only at -400 mesh. Oolite concentrates containing 36.7 to 48.7 wt.% Fe were obtained with heavy liquids but the recovery for a concentrate containing 47.5 wt.% Fe was only 41%.

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INTRODUCTION

The purpose of the mineralogical investigation is to assist beneficiation tests on the Clear Hills iron ore. The work was requested by D.E. Pickett and G.O. Hayslip of the Mineral Processing Division, Mines Branch as part of a joint Federal-Provincial program for assessing the possibilities of up-grading the ore.

Previous detailed investigations have been carried out by D.K. Kidd (1959) of the Research Council of Alberta and by E.H. Nickel et al. (1960) of the Mineral Sciences Division, Mines Branch.

MATERIALS AND METHOD OF INVESTIGATION

Several tons of ore contained in 115 barrels (Table 1) and representing a channel sample of the Clear Hills deposit, Alberta arrived at the Mines Branch on April 18, 1974. Samples for a mineralogical investigation were hand picked from barrels numbered 38, 94 and 127. Polished and thin sections were prepared from these samples and studied under the ore and petrographic microscopes to determine the general features of the ore. The different constituents were identified by a combination of microscopical, X-ray diffraction, differential thermal analysis (DTA), thermogravimetric analysis (TGA), and electron microprobe methods. Electron microprobe and chemical analyses were used to determine the compositions of each constituent: a (image analyzer) "Quantimet" was used to determine grain sizes and relative proportions; and heavy liquids were used to determine specific gravities of individual constituents and to prepare a variety of concentrates.

TABLE 1

Clear Hills Iron Ore

(Channel Sample)

Sample No.	Depth Interval from top of iron bed (in feet)	No. of Barrels
1-21 1	1-5	21
26-46 2	5-10	21
51-71 3	10-15	21
75-93 4	15-19	18
94-96) 5	19-23	3
101-114)		14
116-122) 6	23-27	7
126-135)		10
	Total	115

CHARACTERISTICS OF THE ORE

General Description

The Clear Hills iron ore is a brownish, earthy, friable material consisting of oolites, goethite, siderite, and large silty fragments embedded in a matrix of soft "earthy" material (Figures 1 and 2). Chemical analyses for some of the channel samples were provided by the Alberta Research Council (Table 2), and the data show that the iron content is greatly affected by the moisture content of the ore. In particular the weighted composite sample contains 31.87 wt.% Fe but upon drying 15.48 wt.% water is driven off and the resulting sample contains 37.69 wt.% Fe. An interpretation of TGA data for Sample 38, as received in a sealed container, indicates 16.5 wt.% adsorbed water, 4.5 wt.% absorbed or contained water in goethite, and 3.8 wt.% CO_2 and/or other chemically combined water. We observed that the adsorbed water is lost slowly on exposure to atmospheric conditions and re-adsorbed under humid conditions. Since all of our further

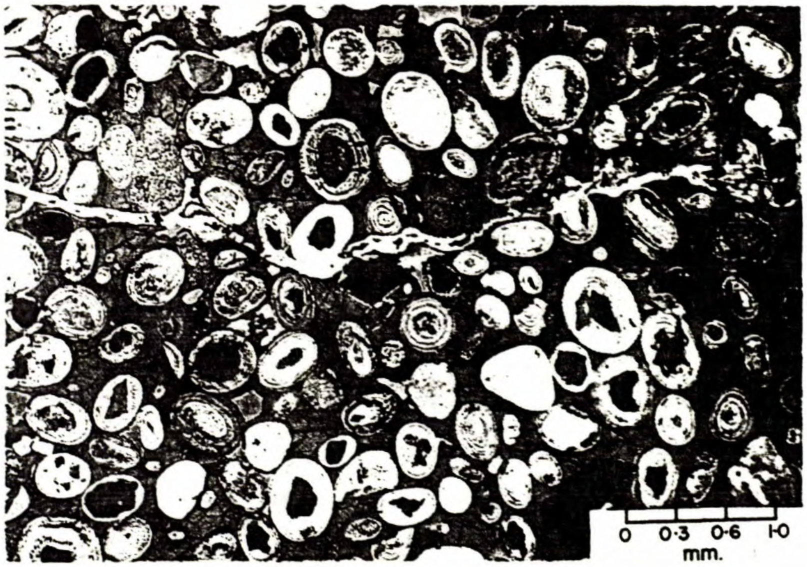

Figure 1. Photomicrograph of the Clear Hills iron ore showing oolites embedded in the "earthy" matrix. The oolites have variable quantities of goethite (white) and some have quartz cores (dark grey). The interstitial earthy matrix has siderite inclusions(light grey) and has many shrinkage fractures. A goethite veinlet (white)is seen in the middle of the photograph.

Figure 2. Photomicrograph of ellipsoidal to spheroidal oolites, some showing a core of angular quartz, others of breccia. The light grey well polished area interstitial to the oolites is siderite and grey one quartz. The groundmass or matrix is the "earthy" material.

investigations were conducted on material that had been exposed to atmospheric conditions it is assumed that most of the adsorbed water had been lost and that samples contained approximately 37 wt. % Fe.

The oolite, matrix, siderite and quartz contents in samples 38 and 94 were determined by quantimet analyses on polished sections (Table 3).

The specific gravity of individual hand specimens and of a pellet prepared from crushed ore varied from 2.74 to 2.78.

TABLE 2

Chemical analysis*of Clear Hills iron ore and iron concentration on wet and dry basis

Elements and Oxides	Sample No. 23R (Wt.%)	48R (Wt.%)	73R (Wt.%)	100R (Wt.%)	123R (Wt.%)	137R (Wt.%)	Weighted Composite
$\%Fe_2O_3$	39.17	29.59	28.24	26.20	33.07	28.82	30.71
%FeO	9.64	17.57	20.63	21.94	10.77	13.83	15.75
$\%P_2O_5$	1.56	1.81	1.31	1.42	1.37	1.42	1.49
$\%SiO_2$	16.59	17.38	17.83	17.31	17.89	19.30	17.7
$\%Al_2O_3$	5.71	5.31	5.25	4.93	4.71	4.73	5.14
%S	0.033	0.024	0.020	0.029	0.025	0.067	0.032
%MnO	0.74	0.63	0.72	1.08	0.90	0.84	0.81
%CaO	1.96	1.64	1.43	1.61	1.85	1.99	1.73
%MgO	0.95	0.85	0.86	1.00	1.11	1.31	1.00
%L.O.I.**	24.25	24.27	24.09	24.02	25.13	28.87	25.01
Total	100.60	99.06	100.38	99.54	96.85	99.18	99.37
%Fe	33.79	32.30	33.32	32.74	30.15	27.96	31.87
%loss of water at 100°C drying	14.41	14.76	15.12	15.24	16.06	17.88	15.48
% Fe in 100°C dried sample	39.48	37.89	39.26	38.63	35.92	34.05	37.69

*Data was supplied by the Alberta Research Council (Hamilton 1974).
**Loss on ignition.

TABLE 3

Compositions of samples in wt.% as determined by Quantimet analyses on polished sections

Constituent	Sample 38	Sample 94
Oolites	63.5	60.0
Matrix	32.0	20.0
Siderite	2.5	14.0
Quartz	2.0	6.0
Total	100.0	100.0

Earthy Matrix

The matrix represents 20 to 32 wt.% of the ore in the samples. It is a pale yellowish material that is isotropic in transmitted light, amorphous to X-ray diffraction, and from microprobe analyses fairly homogeneous in composition. Compositions, as determined by the electron microprobe for two sections and by chemical analyses for a matrix concentrate, are given in Table 4. Its approximate specific gravity, determined by heavy liquids, is 2.0. This material has high Fe and H_2O contents and adsorbs moisture. Some of the moisture escapes under atmospheric conditions, about 10 wt.% moisture is driven of when the sample is heated to 150°C, and the remaining 9.8 wt.% moisture continues to be driven off at a fairly uniform rate to 600°C after which little change was noted up to 980°C.

TABLE 4

Partial analyses of "earthy" matrix

Element or Oxide	Sample 94 Microprobe Wt.%	Sample 38 Microprobe Wt.%	Sample 38 Chemical* Wt.%
Fe (Total)	23.3	23.9	27.64
SiO_2	34.2	35.3	31.70
Al_2O_3	8.2	5.2	6.18
H_2O (3 hrs. at 105°C)			10.01
H_2O (105°C to 980°C)			9.82
C**			3.16
CO_2			0.35

*The sample used for chemical analysis represents the 2.3 float of the -200 +325 mesh fraction which contains 5 to 10 wt.% oolites.

**The C content in this sample is due to adsorbed heavy liquids used in mineral separation.

Oolites

The oolites vary in shape from spheroidal to ellipsoidal (Figures 1 and 2), range from 30 microns to 1 millimeter in diameter, and represent about 60 wt.% of the ore. They generally consist of a concentric arrangement of geothite and earthy material in variable quantities around cores. The goethite contains approximately 55 wt.% Fe (electron microprobe analysis) and 13.9 wt.% absorbed water (calculated from TGA analysis of Sample 38). Its specific gravity was determined through heavy liquid separations as 3.85. The earthy material, hereafter referred to as "oolite gangue", is similar to the earthy matrix in that it is isotropic in transmitted light and amorphous to X-ray diffraction. It has a calculated iron content of 32.1 wt.% and its specific gravity was calculated as 2.50. The oolites cores consist mainly of quartz and goethite, but include siderite, oolite fragments, zircon, and apatite. Some oolites have no distinct cores. Quantimet analysis shows that the oolites contain from 11 to 100 wt.% goethite but that an average oolite consists of 54 wt.% goethite, 42 wt.% oolite gangue, and 4 wt.% quartz.

A partial chemical composition of the oolites, without considering quartz cores, was determined by the electron microprobe using an expanded beam of 25 microns to reduce inhomogeneity effects (Table 5). Quantimet analysis on 10 similar oolites gives a mean mineralogical composition of 48 wt.% goethite and 52 wt.% oolite gangue. The calculated iron content for an average oolite (54 wt.% goethite, 42 wt.% oolite gangue, and 4 wt.% quartz) is 43.2 wt.%.

The specific gravity of the oolites varies from about 2.6 to 3.85 depending upon the goethite content, but for an average oolite it is 3.24

TABLE 5

Electron microprobe analysis of oolites

Element	Sample No. 38 Wt.% (mean of 10 oolites)	Sample No. 94 Wt.% (mean of 10 oolites)	Average Wt.%
Fe (Total)	44.1	42.1	43.1
SiO_2	9.0	10.5	9.7
Al_2O_3	3.7	4.1	3.9

Goethite

Goethite is the main iron-bearing mineral in the ore. It occurs within oolites, mainly as concentric layers 1 to 40 microns wide, and as isolated grains in the matrix. The goethite content in Sample 38 was determined with a Quantimet as 32.1 wt.%. As indicated above the goethite contains 55 wt.% Fe and 13.9 wt.% absorbed water.

Siderite

Siderite occurs throughout the ore, mainly as part of the matrix, interstitial to the oolites, and as inclusions in oolites. Its content varies from 2.5 wt.% to 14 wt.%, with an overall average of probably 6 wt.%.

Phosphorus

Phosphorus is present in minor amounts as a calcium phosphate, tentatively identified as apatite. It was observed in the cores of oolites, as large masses or fragments consisting of brecciated apatite with goethite fillings, and as finely disseminated grains interstitial to the concentric goethite rings of the oolites.

Moisture Content

The samples as received have a moisture content of at least 21 wt.% with 16.5 wt.% as adsorbed water and about 4.5 wt.% as absorbed or contained water related to the goethite. The nature of the adsorbed water is uncertain but it appears to be associated with the earthy matrix and oolite gangue. Some of the water escapes from the samples upon drying or exposure to room temperature. The formation of shrinkage cracks occurring in the matrix material as evidenced in polished sections (Figure 1) suggest a "drying-out". TGA analysis shows a rapid weight loss of 16.5 wt.% up to 175°C, another weight loss of 4.5 wt.% at 385°C the dissociation temperature for goethite, a further weight loss of 3.8 wt. % between 385 and 600°C, and finally a loss of 1.2 wt.% from 600 to 1060°C for a total weight loss of 26 wt.%. An interpretation for the weight loss above 385°C is not readily apparent although some is due to a loss of CO_2 and some may be due to release of other chemically combined water.

Quartz

The quartz occurs mainly as cores but some is present as irregular grains in the earthy matrix.

BENEFICIATION CHARACTERISTICS

Mineralogical properties that may effect the recovery of iron are considered in this section. Since the ore consists of oolites (average Fe content 43 wt.%), matrix (average Fe content 24 wt.%), minor siderite (48.2 wt.% Fe), and minor quartz some of the iron can be recovered by concentrating the oolites and siderite and discarding the matrix and quartz. If full recoveries are achieved the grade of the resulting oolite concentrate would be between 43 and 48.2 wt.% Fe depending on the oolite:siderite ratio. The recovery would depend on the initial grade of the ore,e.g. if the initial ore grade is about 24 wt.% Fe (equivalent to the Fe content of the matrix) there is very little recoverable iron in the form of oolites and siderite, whereas with an initial ore grade of about 43 wt.% Fe most of the iron is recoverable. For sample 38, considered as representative of the ore and assumed to contain 37 wt. % Fe, 77% of the Fe is present as oolites and siderite (maximum possible recovery) and when combined the concentrate would have an Fe content of 43 wt.%. An approximate relationship between initial ore grade and possible recovery as an oolite concentrate is given in Figure 3.

Size analyses were performed with a Quantimet on the oolites, siderite, and quartz to determine the size distribution of each constituent (Table 6 and Figure 4). These data show that 82% of the oolites and 15% of the siderite are larger than 147 microns in diameter (100 mesh) and would be liberated if the ore was ground to -100 mesh. If all were recovered it would represent a recovery of 60% at a grade of 43 wt.% Fe. Since crushed ore always contains some fine material a portion of the fine oolites and remaining siderite will also be free. Screen analyses were performed on the ore ground to -20, -28, -65, and -100 mesh to determine the quantities of fine material in ore crushed to each size (Table 7 and Figure 4). By combining the Quantimet size analysis with the screen analyses, the per cent

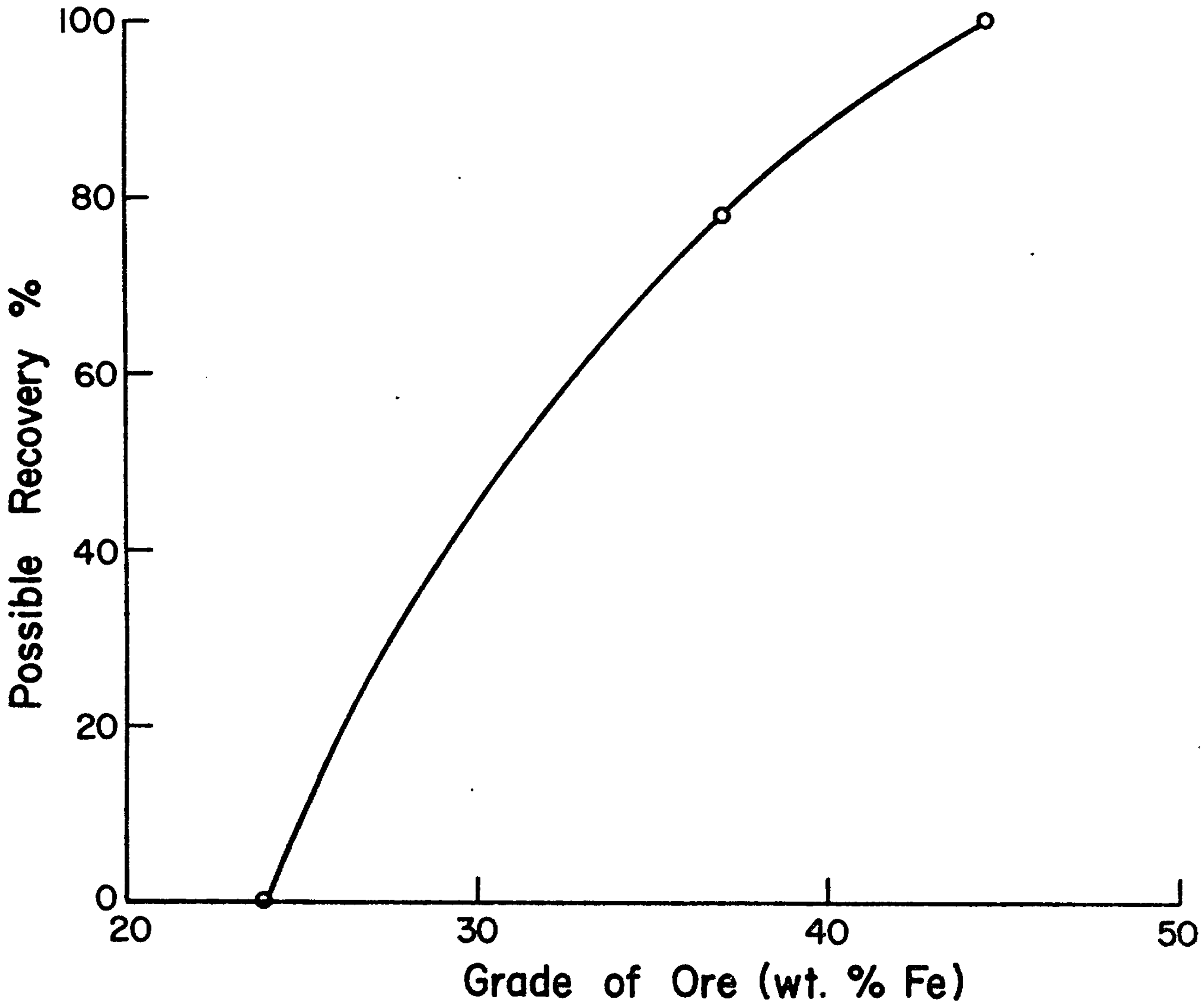

Figure 3. Approximate relationship between grade of dried ore and possible recovery as an oolite concentrate.

recoverable Fe was determined for -20, -28, -65 and -100 mesh fractions (Table 8). The results show that to recover most of the oolites, the ore must be ground to -65 mesh. Finer grinding will not significantly improve liberation of the oolites.

TABLE 6

Size distribution of Oolites, Siderite and Quartz in Sample 38

Size (Mesh)	Oolites Wt.%	Siderite Wt.%	Quartz Wt.%
-400	0	40	13
200 to 400	4	25	15
150 to 200	7	(	(
100 to 150	7	(20	(24
28 to 100	82	15	48
Total	100	100	100

TABLE 7

Screen analyses for sample 38

-28 Mesh		-28 Mesh		-65 Mesh		-100 Mesh	
Mesh Size	Distribu- tion Wt.%	Mesh Size	Distribu- tion Wt.%	Mesh Size	Distribu- tion Wt.%	Mesh Size	Distribu- tion Wt.
20to35	42.1						
35 to65	30.8	28to65	65.3				
65to100	5.8	65to150	15.2	65to100	9.5	100to150	15.8
-100	21.3	150to325	14.8	100to200	29.2	150to200	21.8
		-325	4.7	200to325	18.5	200to325	19.1
				325to400	5.0		
				-400	37.8	-325	43.3
Total	100.0		100.0		100.0		100.

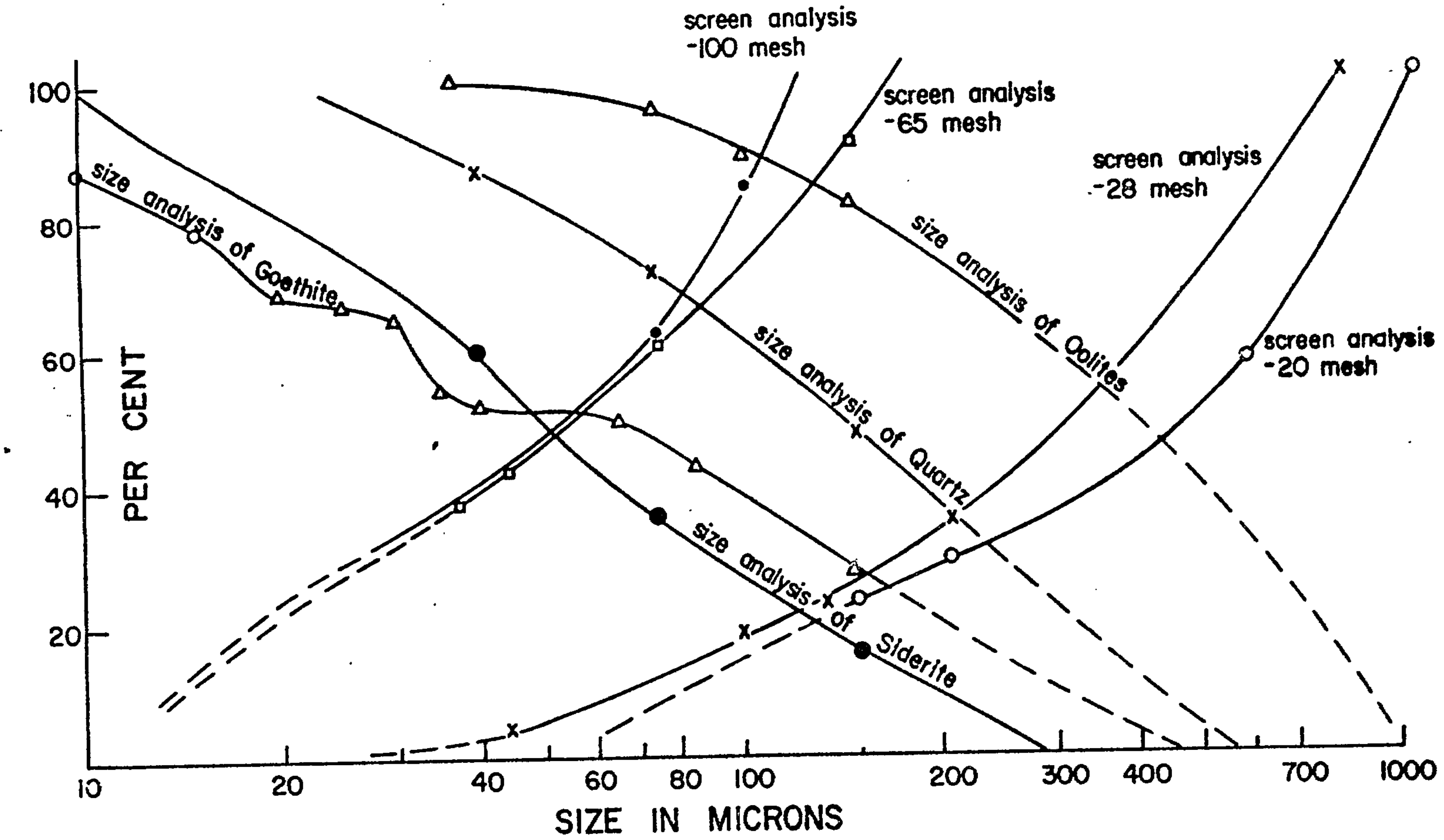

Figure 4. Size distribution of oolites, quartz, siderite, and goethite plotted as per cent larger than, and size distributions of screened fractions plotted as per cent finer than.

TABLE 8

Per cent of Recoverable Iron as Liberated Oolites and Siderite

(determined by Combining Quantimet Size Analysis and Screen Analysis)

	Size Fractions			
	-20 Mesh	-28 Mesh	-65 Mesh	-100 Mesh
Liberated Oolites(%)	46	57	93	95
Liberated Siderite(%)	15	17	52	56
Recoverable Fe as oolites and siderite(%)	~~[illegible]~~ 37	~~41~~ 51	71 [illegible]	~~72~~ [illegible]

It was observed that the oolites are more resistant to comminution than the matrix and that the proportion of oolites to matrix is higher in coarser screen fractions than in finer ones of a crushed ore (Table 9). Some of the oolites in the coarser fractions, however, still contain quartz cores and grinding to perhaps -150 mesh may be required to remove this remaining quartz. The oolites in the finer fractions (150 to 325 and -325 mesh) are broken but oolite fragments with wide goethite layers as well as massive goethite tend to concentrate in the 150 to 325 mesh fraction, whereas oolite fragments with narrow goethite layers tend to concentrate in the -325 mesh fraction. It was estimated, however, that the goethite contents are similar in both the 150 to 325 and -325 mesh fractions.

TABLE 9

Distribution of Constituents in Fractions of Sample 38 ground to -28 mesh, Screened, and Separated with Heavy Liquids

Fraction (Mesh)	2.6 float (mainly matrix) Wt.%	2.9 float (oolites + matrix) Wt.%	2.9 Sink (oolites) Wt.%	Total
28 to 65	8.5	39.7	16.6	64.8
65 to 150	6.0	2.3	6.8	15.1
150 to 325	10.1	0.7	3.8	14.6
-325	3.8	1.1	0.6	5.5
Total	28.4	43.8	27.8	100.0

A series of tests were performed to determine whether oolite concentrates can be obtained from this ore by separating 20 to 35, 28 to 65, 25 to 150, and 150 to 325 mesh fractions with heavy liquids and analysing polished sections of the resulting subfractions with a Quantimet. The data for the 20 to 35 mesh fraction (Table 10) show that it is possible to obtain oolite concentrates (subfractions) containing as little as 36.7 wt.% Fe to as much as 48.7 wt.% Fe. The calculated grade for the combined oolite subfractions (float 3.1 to sink 3.8) is 42 wt.% Fe and the calculated recovery is 81.5 %. The high recovery for this coarse closely sized fraction is due to concentration of oolites in the coarse fraction.

TABLE 10

Heavy Liquid Seperation of -20 +35 mesh Fraction

Subfraction	Distribution Wt.%	Goethite Content (Quantimet analysis)	% Fe(Calc.)	Constituents
Float 2.3	2.8	6.8	27.0	Matrix and Oolites
Float 2.5	6.5	6.8	27.0	Matrix and Oolites
Float 2.6	8.0	5.0	26.6	Matrix and Oolites
Float 2.9	8.1	18.4	30.5	Matrix and Oolites
Float 3.1	21.4	20.4	36.7	Oolites
Float 3.3	8.8	48.9	41.3	Oolites
Float 3.5	19.2	62.2	46.1	Oolites
Float 3.7	22.9	53.3	44.3	Oolites
Float 3.8	1.9	71.3	48.7	Oolites
Sink 3.8	0.4	--	--	Oolites
Total	100.0			

Another test was performed on a sample ground to -65 mesh to obtain some idea of the type of concentrate that would be obtained by concentrating all grains heavier than those having a specific gravity of 3.33. Sized fractions of the sample were separated into sink and float subfractions with a heavy liquid (Sp. Gr. = 3.33) and analysed chemically (Table 11). The calculated grade for the combined sink fractions (-65 mesh) is 47.49 Wt.% Fe and the recovery is 41%.

TABLE 11

Fe Analyses for sink/float Subfractions of Sample 38 using 3.33 Sp.Gr.Liquid

Mesh Size	Distribution (Wt. %)		Fe Contents (Wt.%)		SiO_2 (Wt. %)	Insol. (Wt. %)
	Sink	Float	Sink	Float	Sink	Sink
65 - 100	2.7	6.9	46.11		8.31	8.53
100-200	10.1	19.1	47.43			
200-325	6.8	11.7	47.83		8.54	8.72
325-400	1.5	3.5	48.00	29.21		
-400	10.8	26.9	47.71	33.17	7.71	7.89
Total	31.9	68.1				

A test was conducted to evaluate recoveries and concentrate grades that could be obtained from this ore crushed to -28 mesh. The crushed ore was screened into fractions,separated into subfractions with heavy liquids, and the mineralogical composition of each subfraction was estimated with an ore microscope. The iron contents, grades, and recoveries were calculated (Table 12). It is considered noteworthy that in this test we obtained a recovery of 33.5% at a concentrate grade of 44.5 Wt.% Fe. Table 8 shows that for this ore ground to -28 mesh the maximum recovery is 41% at a grade of about 43 Wt.% Fe.

TABLE 12

Heavy Liquid Separations of Sample 38 Crushed to -28 mesh and Calculated Recoveries and Grade of Concentrate

	Sp. gr. = 2.6		Sp. gr. = 2.9		Sp. gr. = 3.33	
	Sink Wt.%	Float Wt.%	Sink Wt.%	Float Wt.%	Sink	Float
28to65 mesh	56.16	8.51	16.56	48.11	0.05	64.62
65to150 mesh	9.09	5.99	6.78	8.30	4.36	10.72
150to325 mesh	4.62	10.01	3.86	10.77	1.41	13.22
-325 mesh	1.78	3.84	0.64	4.98	0.00	5.62
Total	71.65	28.35	27.84	72.16	5.82	94.18
Fe content (Calc.) (Wt.%)	24.5		12.4		2.8	
Grade (Wt.% Fe) (Calc.)	34.0*		44.5		48.0	
Recovery (%) (Calc.)	66.0		33.5		8.0	

*Quartz was concentrated in this fraction and had the effect of reducing the grade.

The possibility of recovering a goethite concentrate rather than on oolite concentrate was also considered. Sample 38 contains 32 wt. % goethite (Quantimet analysis) which represents 48% of the Fe in the ore. Size analysis data for goethite (Table 13 and Figure 4) show that 55 wt.% of the goethite occurs as bands narrower than 40 microns (≃-400 mesh) with most being narrower than 15 microns. On the other hand 45 wt.% of the goethite is larger than 65 microns (≃+275 mesh) and 28 wt.% larger than 150 microns (+100 mesh). The proportions of Fe in the ore as +275 mesh and +100 mesh goethite are 22 and 13% respectively. If this coarse goethite can be recovered the resulting grade of the concentrate could be as high as 55 wt.%. No pure goethite concentrates however, were obtained in the tests with heavy liquids.

A graph showing maximum possible recoveries vs concentrate grades for this ore as interpreted from this study is given in Figure 5.

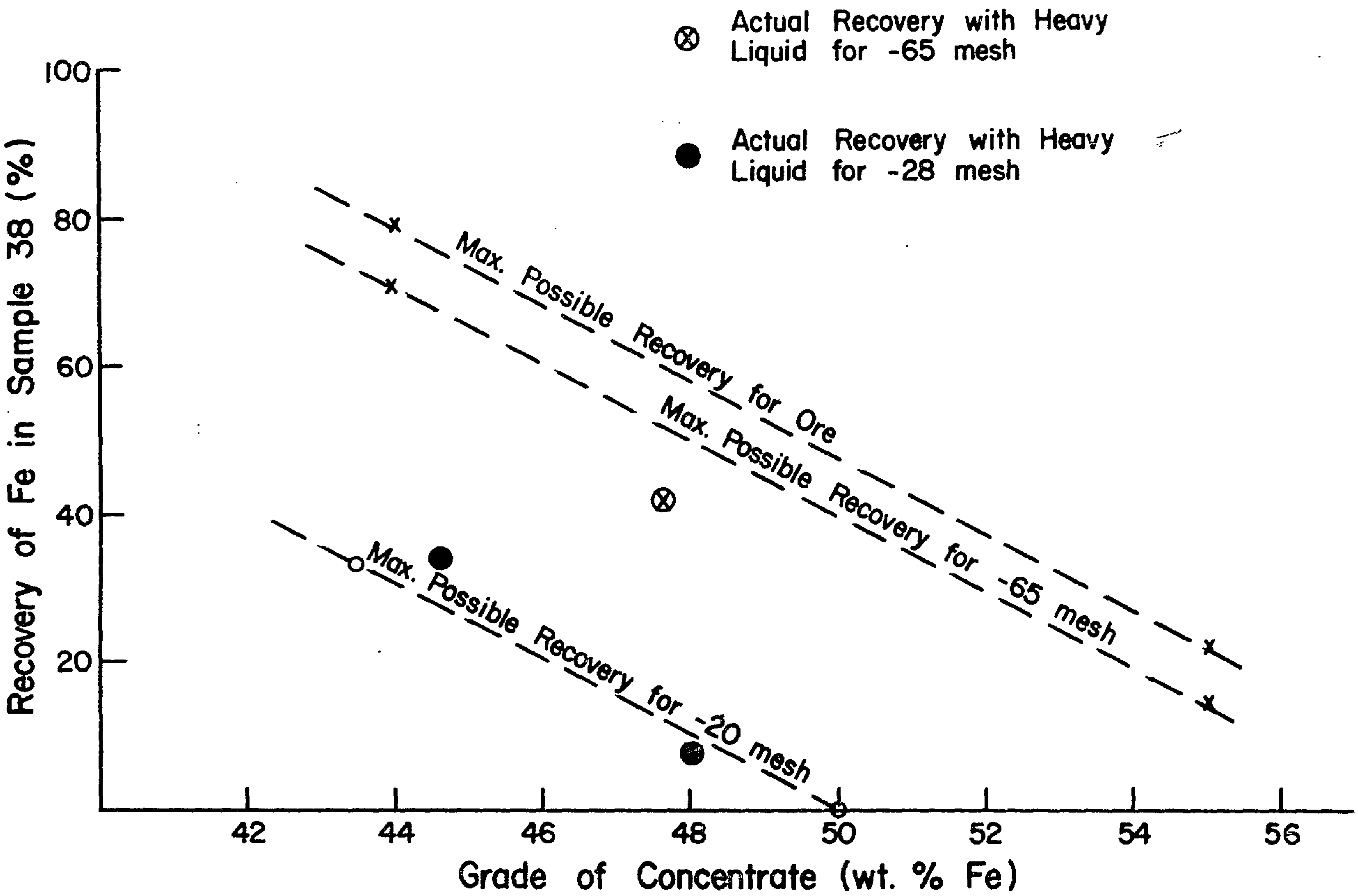

Figure 5. Relationship between maximum recovery of iron in sample 38 and concentrate grade. The maximum possible recoveries were calculated from Quantimet size analyses data.

TABLE 13

Distribution of Fe According to Size Analyses of Goethite

Size (microns)	Wt. % Goethite	Wt. % Fe (Calc.)*
0-5	16.8	8.1
5-10	9.1	4.4
10-15	9.9	4.6
15-20	1.7	0.8
20-25	1.8	0.9
25-30	10.8	5.2
30-35	2.2	1.1
35-40	2.6	1.2
40-65	0.0	0.0
65-70	6.6	3.2
70-85	0.0	0.0
85-90	10.5	5.1
90-150	0.0	0.0
150-500	28.0	13.4
Total	100.0	

*On the basis of 48 wt.% of Fe in the ore occurring as goethite.

CONCLUSIONS AND RECOMMENDATIONS

1. The ore consists largely of oolites and an iron-bearing matrix, and contains minor amounts of siderite, quartz, and a phosphate (apatite ?).
2. It also contains large quantities of adsorbed water which escapes to the atmosphere in dry conditions and is re-adsorbed under humid conditions. This significantly affects analytical data. It is recommended that all test work be done on ore dried at 100°C.
3. It is considered that the only practical method of upgrading this ore is by concentrating the oolites and siderite and discarding the matrix and quartz. The maximum recovery possible for the sample studied is 77% and the grade of the

concentrate 43 wt. % Fe. A concentrate of 47.49 wt. % Fe was obtained but with a recovery of 41%.

4. It is not practical to produce a goethite concentrate from this ore because the maximum recovery as a goethite concentrate from the sample studied is 22%.
5. Size analysis data show that the ore must be ground to -65 mesh to liberate 93% of the oolites and that grinding finer does not significantly increase oolite liberation.
6. The oolites are more resistant to comminution than the matrix. This suggests that recoveries might be enhanced by grinding the ore to -20 mesh, recovering the oolites, regrinding the tails to -65 mesh, and recovering the remaining oolites and siderite.
7. Some of the oolites in an oolite concentrate obtained by the above method would still contain quartz cores and would have to be ground to at least -150 mesh to liberate most of the quartz.

ACKNOWELDGEMENTS

The authors gratefully acknowledge the support of personnel in the Mineral Sciences Division in undertaking this investigation. These include J. Cloutier and J. Hole for the chemical analyses; W. Bowman for the DTA and TGA analyses; E.J. Murray for the X-ray diffraction analyses, Y. Bourgoin for performing the heavy liquid separations and P. O'Donovan and Y. Bourgoin for preparing the polished sections.

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WP/DCH/RGP/pg