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Non-Conventional Beneficiation of Hydrated Iron Oxide Ores

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Abstract: An earthy hydrated iron oxide brownish black coloured lumpy sample was collected for process evolution studies for metallurgical industry assaying minimum 62%Fe. The sample assayed 57.65% Fe (T), 4.40% SiO2, 2.69% Al2O3, and 10.81% LOI containing mainly H2O. The sample contained mainly goethite with subordinate amount of ferruginous clay. It contained minor amount of hematite with trace amounts of quartz, gibbsite and magnetite. The sample is not amenable to particle size refining, gravity and magnetic separation, hence as desired +62% Fe grade concentrate could not be produced, due to hydrous nature of iron ore mineral. The process of calcinations at -10mm size, water quenching followed by wet screening process at 3 and 0.2mm yielded: (a) An hard chips iron concentrate (-10 +3 mm) assaying 63.00% Fe, 3.84% SiO2, 2.26% Al2O3, 3.98% LOI and 78% Tumbler Index with 35% Fe distribution, at wt.% yield of 33.0 for use in sponge iron industry.(b) A sandy concentrate (-3 +0.2 mm) assaying 64.12% Fe, 3.34% SiO2, 2.06% Al2O3, 3.86% LOI with 54.5% Fe recovery at 50.0 wt. % yield meeting the sinter industry specifications. (c) The slimes assaying 62.50 %Fe 4.04% SiO2, 2.56% Al2O3, and 4.08% LOI may be used in pellet industry as it is. Alternatively, further processing of slimes using WHIMS could yield a DR grade concentrate assaying 67.14% Fe, 1.56% SiO2, 1.16% Al2O3, 1.40%LOI with 8% Fe distribution at wt. % yield of 7 for use in DR pellet industry, while slimy non magnetic tails assaying 50% Fe may be used in cement industry. The evolved process offers scope for using the hydrated iron oxide ores in metallurgical and cement industry with nil discharge.

Key words; Goethite, calcinations, WHIMS, gravity concentration

INTRODUCTION

Review of Indian steel industry reveals that the steel output is expected to grow three fold during the year 2020. This warrants about 50 million tons of quality iron lumps or beneficiated agglomerates during the year 2020 [1-3]. With increase in demand for quality lumpy ores, the ratio of lumps to fines in mines tend to reverse from existing 2:1 ratio to 1:2.

Further, increased LOI content in pellet grade concentrate due to hydrated iron oxides may cause bottlenecks in conventional Pelletization warranting modifications. Also due to soft, brittleness, relatively low specific gravity and low magnetic susceptibility nature of hydrated iron oxides [HIO] as compared to anhydrous iron oxide minerals, most of it is lost in tails in conventional iron ore processing plants as reported by previous works.[1 &2, 4-10] The low iron content due to chemically bound water in lattice and fine interlocking of clayey minerals, the hydrated iron oxide ores are mostly used in cement industry as they are not accepted in metallurgical industry. The aim was to evolve a process for HIO for use in metallurgical industry with Fe >62%.

EXPERIMENTAL

Material: About 250 kgs of hydrated iron oxide lumpy sample was collected from CNHalli, Tumkur for process evolution studies with the aim of utilizing the product in high revenue yielding metallurgical industry. Since the grade was low and sample had tumbler value of 50% it could not be used in metallurgical industry, however it was used in cement industry as iron additive.

Equipment:250 x150mm Roll crusher, Carpco riffler, Tyler sieve set, Electrical Muffle furnace, MPE 175 x 350 mm tumbling mill with 13.5 kg 25 mm steel balls/ 19 mm to37.5mm steel rods MPE Laboratory diagonal deck table, Mozley 50 mm cyclone test rig, CE 4x2 lab WHIMS, , pan filter, drying oven and balances.

Methods: Standard feed preparation and sampling methods, laboratory testing methods, mineralogical and assay methods enumerated by hand books were followed. The experimental work has been categorized as characterization, Calcination studies, and other auxiliary tests.

RESULTS AND DISCUSSION

Characterization studies: The as received sample consisted of brownish black colored lumps with considerable amount of brown coloured fines. The bulk density of the sample was 1.8 t/m^3. The sample assayed 57.65% Fe (T), 0.19% FeO, 4.40% SiO_2, 2.69% Al_2O_3, 0.19% Na_2O, 0.06% K_2O, 0.13% P_2O_5, 0.14% Mn 10.20% H_2O and 10.81% LOI. The sample contained mainly goethite with subordinate amount of ferruginous clay. It contained minor amount of hematite with trace amounts of quartz, gibbsite and magnetite. Goethite is present as spherolitic aggregates and banded masses. Cavities in the goethite are filled with clay. Hematite is fine grained (2 to 100 microns) and present as discrete grains / inclusions within the goethite. It is altering to goethite along the grain boundaries and cleavage planes. Clay is normally present in the cavities of goethite. Diagnostic process amenability test of the sample at -65 mesh size deslimed over 325 mesh, by Sink-Float test at3 SG using TBE and Frantz Iso dynamic test could yield concentrates assaying 60-61 % Fe failing to meet the specifications of metallurgical industry [Fe >62 concordant with the results of previous works on characterization of iron ores. [4 & 6] Hence it was decided to try the amenability of sample to pyro processing.

Preliminary tabling and WHIMS tests varying Mesh of Grind: Tabling tests were conducted on samples rod mill stage ground to -30, -48 -65 and -100 mesh (d_{80} 310, 210, 160 and 110 microns respectively). Optimum results were obtained at -48mesh (d_{80} 210 microns) yielding concentrate assaying 61.71% Fe, 1.25% SiO_2, 1.60% Al_2O_3 and 7.71% LOI. The concentrate contained mainly goethite. Wet high intensity magnetic separation (WHIMS) tests were conducted on samples rod mill stage ground to -30/-48/-65 and -100 mesh (d_{80} 310, 210,160 and 110 microns respectively). Optimum results were obtained at -30 mesh grind, d_{80} 310 microns producing concentrate assaying 59.31% Fe with 64.1% Fe distribution at 62.1 wt. % yield. The concentrate contained mainly goethite. The findings are similar to that obtained Diagnostic process amenability tests [4 and 6] and previous findings on anhydrous and hydrous iron oxides[7-10] Hence it was decided to opt for pyro processing tests to remove 10.20% water combined in lattice for up-gradation.

Calcination tests varying size: The proceeding gravity and WHIMS tests could not produce concentrate assaying + 62.5% Fe and meeting the size requirement of either pellet or sinter grade. The mineralogical and diagnostic study indicated that the sample contained hydrated iron oxide only, indicating chemical processing is the rate. The literature indicated that goethite start dehydrating when heated at temperature above 230°C. Calcination tests were conducted on as received samples stage crushed to-20 mm/-10 mm/ -2mm followed by water quenching and screening over 65 mesh and 300 mesh. The results are given in Table 2 The size analysis of feed is given in Table 1. The results indicated that optimum results were obtained at -10 mm size yielding a composite calcined product assaying 64.22% Fe, 2.93% SiO_2, 1.22% Al_2O_3 and 3.28% LOI. The calcined product upon screening over 65 and 300 mesh yielded a sinter grade concentrate assaying 64.19% Fe with 92.7% Fe distribution meeting the requirements of the party. The slime rejects may further be concentrated to pellet grade, enhancing the overall recovery. Deviation from 10 mm size significantly affects the results.

Table 1: Size analysis data

Size in mm	**Cum. Wt% U/S**		
	-20 mm	**-10 mm**	**-2mm**
20	100.0	100.0	100.0
15	90.2	100.0	100.0
12	88.6	100.0	100.0
10	82.0	100.0	100.0
6	70.0	82.0	100.0
4	60.0	65.0	100.0
2	50.0	60.0	100.0
1	30.0	52.0	70.0
0.5	21.0	30.0	40.0
0.2	10.0	18.0	20.0

Effect of calcinations temperature: The calcination, water quenching and screening tests were conducted varying calcinations temperatures. The test result is given in **Table 3**.Increase in temperature increases the grade. A minimum of 350°C is essential to get the stipulated grade. The tests at 350°C yielded a sinter grade concentrate assaying 64.19% Fe with 92.7% Fe distribution meeting the specification of the party. The review of literature on DTA-TGA indicated that goethite decomposed to hematite at 300°C [4].

Table 2: Calcination test varying size

Conditions : 1 kg as received sample stage crushed to -20 mm/-10 mm/-2 mm And subjected to calcinations at 350°C, 1 hour retention time, ½ hour drying and preheating calcined product water quenched and screened over 10 mm, 65 mesh and 300 mesh

Results:

Size	Product	Wt.%	Fe(T)	
			Assay%	%Dist
-10 mesh (-2 mm) d_{80} 1.1 mm	-10 mm +65 mesh	72.9	62.28	76.3
	-65 +300 mesh	15.0	60.83	15.0
	-300 mesh	9.0	55.23	8.7
	Loss	6.0	--	--
	Head (Calc.)	**100.0**	**59.50**	**100.0**
	Calcined Product (Calc.)	**94.0**	**60.77**	**100.0**
-10 mm d_{80}	-10 mm +65 mesh	85.8	64.19	92.7
	-65 +300 mesh	4.3	65.97	4.8
	-300 mesh	2.3	62.10	2.4
	Loss	7.6	--	--
	Head (Calc.)	**100.0**	**59.34**	**100.0**
	Calcined Product (Calc.)	**92.4**	**64.22**	**100.0**
-20 mm d_{80}	-20 +6 mm	40.7	63.75	44.5
	-6 mm +65 mesh	40.7	63.83	44.5
	-65 +300 mesh	6.9	59.57	7.0
	-300 mesh	4.4	52.09	4.0
	Loss	7.3	--	--
	Head (Calc.)	**100.0**	**58.33**	**100.0**
	Calcined Product (Calc.)	**92.7**	**62.92**	**100.0**

Calcination tests varying time: Calcination tests were conducted at 350°C varying time from 0.5, 1 and 2 hours followed by water quenching and screening. The test conditions and results are given in Table 4 . The tests indicated that calcined product grades increases with calcinations time and saturated at time 2 hours. A minimum of 1 hour calacination at temperature >350°C is essential for achieving grade.

Final test: The process of calcinations at -10 mm size, water quenching followed by wet screening process at 3 and 0.2mm. The results are given in Table 5. The above process produced

(a) An hard chips iron concentrate (-10 +3 mm) assaying 63.00% Fe, 3.84% SiO_2, 2.26% Al_2O_3, 3.98% LOI and 78% Tumbler Index with 35% Fe distribution, at wt.% yield of 33.0 for use in sponge iron industry.

(b) A sandy concentrate (-3 +0.2 mm) assaying 64.12% Fe, 3.34% SiO_2, 2.06% Al_2O_3, 3.86% LOI with 54.5% Fe recovery at 50.0 wt. % yield meeting the sinter industry specifications.

(c) The slimes assaying 62.50 %Fe 4.04% SiO_2, 2.56% Al_2O_3, and 4.08% LOI with 10.5 % Fe distribution at wt.% yield of 10 may be used in pellet industry as it is. Alternatively, further processing of slimes using WHIMS could yield a DR pellet grade concentrate assaying 67.14% Fe, 1.56% SiO_2, 1.16% Al_2O_3, 1.40%LOI with 8% Fe distribution at wt. % yield of 7 for use in pellet industry, while slimy non magnetic tails assaying 50% Fe may be used in cement industry. The evolved process offers scope for using the hydrated iron oxide ores in metallurgical and cement industry with nil discharge leading to sustainable development of the region. The above process may also be attempted for other volatile impurity bearing iron oxides.

Table 3: Calcination test varying temperature

Conditions: 1 kg -10 mm stage crushed as received sample, calcined for ½ hr pre-drying pre-heating and 1 hour soaking retention time at varying temperatures of 250 ^oC, 350^oC and 450^oC hot calcined product was water quenched and screened over 65 and 300 mesh sieves

Results:

Temperature ^oC	Product	Wt.%	Fe(T)	
			Assay%	%Dist
250 ^oC	-10 mm +65 mesh	82.4	63.12	87.6
	-65 +300 mesh	7.7	64.08	8.3
	-300 mesh	4.1	59.23	4.1
	Loss	2.8	--	--
	Head (Calc.)	**100.0**	**59.37**	**100.0**
	Calcined Product (Calc.)	**97.2**	**61.08**	**100.0**
350^oC	-10 mm +65 mesh	85.8	64.19	92.7
	-65 +300 mesh	4.3	65.97	4.8
	-300 mesh	2.3	62.10	2.4
	Loss	7.6	--	--
	Head (Calc.)	**100.0**	**59.34**	**100.0**
	Calcined Product (Calc.)	**92.4**	**64.22**	**100.0**
450^oC	-10 mm +65 mesh	84.0	64.76	90.8
	-65 +300 mesh	5.7	63.00	6.0
	-300 mesh	3.1	61.04	3.2
	Loss	--	--	--
	Head (Calc.)	**100.0**	**59.88**	**100.0**
	Calcined Product (Calc.)	**92.8**	**64.53**	**100.0**

Table 4: Calcination test varying time

Conditions: 1 kg -10 mm stage crushed as received sample, calcined at 350°C, calcinations time varying from 1/2, 1 and 2 hours. Hot claimed product was water quenched and wet screened over 65 and 300 mesh sieve

Results:

Time in hours	Product	Wt.%	Fe(T)	
			Assay%	%Dist
½ hr	-10 mm +65 mesh	83.0	63.67	89.5
	-65 +300 mesh	6.4	62.67	6.8
	-300 mesh	3.7	59.05	3.7
	Loss	6.9	--	--
	Head (Calc.)	**100.0**	**59.04**	**100.0**
	Calcined Product (Calc.)	**93.1**	**63.41**	**100.0**
1 hr	-10 mm +65 mesh	85.8	64.19	92.7
	-65 +300 mesh	4.3	65.97	4.8
	-300 mesh	2.3	62.10	2.4
	Loss	7.6	--	--
	Head (Calc.)	**100.0**	**59.34**	**100.0**
	Calcined Product (Calc.)	**92.4**	**64.22**	**100.0**
2 hrs	-10 mm +65 mesh	84.6	64.43	92.2
	-65 +300 mesh	4.6	62.35	4.9
	-300 mesh	2.8	62.25	2.9
	Loss	8.0	--	--
	Head (Calc.)	**100.0**	59.12	**100.0**
	Calcined Product (Calc.)	**92.0**	**64.27**	**100.0**

Table 5: Final Test

Conditions: As received sample stage crushed to -10 mm, calcined at 450°C for ½ hours drying and pre-heating. Hot calcine was water quenched and wet screened over 3 & 0.2mm

Optional; -0.2mm slimes subjected to WHIMS 6mm ball matrix, 1Tesla open air intensity

Results:

Product	Wt.%	Fe(T)	
		Assay%	%Dist
-10+3 mm chips concentrate	33.0	63.00	35.0
-3+0.2 mm sandy concentrate	50.0	64.12	54.5
-0.2mm WHIMS mag concentrate	7.0	67.14	8.0
0.2mm WHIMS non mag co- product	3.0	50.00	2.5
Loss on calcination	7.0	--	--
Head (Calc.)	**100.0**	**59.10**	**100.0**
Calcined Product (Calc.)	**93.0**	**63.48**	**100.0**
-10 mm +0.2mm sand[Calc]	83.0	63.67	89.5
-0.2mm slimes[Calc]	10.0	62.50	10.5

CONCLUSION:

An earthy hydrated iron oxide brownish black coloured lumpy sample with 50% tumbler index, assaying 57.65% Fe (T), 4.40% SiO_2, 2.69% Al_2O_3, and 10.81% LOI containing mainly H_2O was collected for process evolution studies for metallurgical industry assaying minimum 62%Fe. The process of calcinations at -10 mm size, water quenching followed by wet screening process at 3 and 0.2mm yielded (a) An hard chips iron concentrate (-10 +3 mm) assaying 63.00% Fe, 3.84% SiO_2, 2.26% Al_2O_3, 3.98% LOI and 78% Tumbler Index with 35% Fe distribution,at wt.% yield of 33.0 for use in sponge iron industry. (b) A sandy concentrate (-3 +0.2 mm) assaying 64.12% Fe, 3.34% SiO_2, 2.06% Al_2O_3, 3.86% LOI with 54.5% Fe recovery at 50.0 wt. % yield meeting the sinter industry specifications.(c) The slimes assaying 62.50 %Fe 4.04% SiO_2, 2.56% Al_2O_3, and 4.08% LOI may be used in pellet industry as it is. Alternatively, further processing of slimes using WHIMS could yield a DR grade concentrate assaying 67.14% Fe, 1.56% SiO_2, 1.16% Al_2O_3, 1.40%LOI with 8% Fe distribution at wt. % yield of 7 for use in DR pellet industry, while slimy non magnetic tails assaying 50% Fe may be used in cement industry. The evolved process offers scope for using the hydrated iron oxide ores in metallurgical and cement industry with nil discharge leading to sustainable development of the region. The above process may also be attempted for other volatile impurity bearing iron oxides.

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