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Recovery of iron minerals from low-grade iron ore tailings by conducting beneficiation studies using hydrocyclone

Detailed mineralogical and geochemical studies are carried out on iron ore tailings from Donimalai iron ore deposits (DIOD), India. The mineralogical studies using X-ray powder diffraction on the tailing samples shows the presence of major iron (Fe) bearing phase hematite (Fe_2O_3) and the gangue minerals such as alumina and silica are present in the form of kaolinite ($Al_2Si_2O_5(OH)_4$) and gibbsite ($Al(OH)_3$). The geochemical study of the tailings shows the assay amount up to 56.32% Fe, 9.35% SiO_2, 5.82% Al_2O_3, 0.057% P and 2.60% loss on ignition (LOI). An elaborate study on the tailings was carried out with the help of DLS (diffraction light scattering system), SEM-EDS (scanning electron microscope-energy dispersive spectroscopic analysis) which shows that most of the slime particles lie below the range of 150μm to 70μm, only less number of fractions are found in the lesser micron size. Moreover, tailing contains Fe (iron) bearing hematite with interlocking gangue minerals. The detailed liberation analysis of the tailings shows the major iron-bearing phase has been present in coarser size and more gangue minerals are present in the finer fractions. Beneficiation is carried out for the tailings using hydrocyclone. Based on selection of different process parameters such as spigot diameter (mm), vortex finder diameter (mm), pressure (psi), and solid concentration (%) the beneficiation is performed. Beneficiation through hydrocyclone increases the Fe content from 56.32 to 62.04% with a solid recovery rate of 32%, which can be utilized for blast furnace operation.

Kdywords: *Donimalai, tailing, XRD (X-ray powder diffraction), SEM-EDS (scanning electron microscope-energy dispersive spectroscopy), DLS (dynamic light scattering), hydrocyclone.*

1. Introduction

Indian iron ore deposits are classified into five major groups based on its origin and occurrence, in which banded iron formation (BIF) of Precambrian iron ore series is considered to be the largest deposits of all, followed by titaniferrous and vanadiferous deposits (Pichamuthu 1974, Murthy and Chattterjee 1995). Major ore minerals of banded iron formation of Indian iron ore series are hematite and magnetite which is considered to be the major feed stock material for iron making in blast furnace operation. With a total resource of 28.52 billion tonnes of hematite and magnetite, India is one of the leading producers of marketable iron ore, in which hematite is distributed majorly (i.e. around 62.68% of total resource) in India as compared to magnetite (Yellishetty, Ranjith et al. 2010). Banded iron formation (BIF) of Indian iron ore shows characteristics of massive, laminated, shaly ore with high iron (Fe) content. Iron ores of India are quite soft and friable in nature and generate significant amount of fines during ore dressing and handling (Pradip 2006). These fines are relatively low grade and cannot be utilized directly in a blast furnace for iron making, because it affects the blast furnace productivity due to the presence of high amount of alumina and silica in the feed, so it has to be disposed of as waste in tailings pond. An estimate of around 32 per cent of the mined ores ends up as tailings (Ghose and Sen 2000). The tailings leads major environmental problems such as degradation of soil quality and pollution in surface and ground water (Rudramuniyappa 1997). Which leads to raise questions on safe disposal strategies and waste management. Tailings which are accumulated as waste can be a potential resource after the depletion of available rich resources.

The major problem in utilizing the fines directly for blast furnace operation is its high gangue content and its physical characteristics. The size range and soft nature of the fines make the beneficiation process complicated (Pradip 1994). Higher gangue content (i.e. higher alumina and silica content) which reduces the sintering strength, not only that if the fines are fed directly without beneficiation which leads to poor productivity and large amount of slag formation (Kumar and Mukherjee 1994). More the gangue content in the fines, which reduces the RDI (reduction degradation index) value of sinters and pellets in blast furnace operation (Lu, Holmes et al. 2007). Higher the gangue minerals is then higher will be flux and coke requirement in blast furnace operation. By reducing the gangue minerals the coke and flux consumption

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for blast furnace will be reduced and leads to higher productivity.

Till date several research work has been carried out for beneficiating the low grade iron ore fines, in order to utilize it for blast furnace operation (Mukherjee, Pan et al. 2006, Roy, Das et al. 2007, Jyoti, Rath et al. 2010). But till date it is not a common practice of introducing several characterization techniques to study the mineralogical characteristics of the fines, only a very few research has been carried out in the field of mineralogical characterization. The current research work tries to fill a gap which has been left by the predecessors on the field of the detailed qualitative and quantitative analysis. Such studies will increase our knowledge about the mineralogical characteristics of iron ore fines. Determination of the mineralogy of iron ore particles can be carried out by several methods – indirect or direct measurements. Usually indirect measurement assumes that the minerals in the examined ore are stoichiometric (Zhang. N. 2001., Benson.S 2001) and standard chemical analysis of iron ore and its constituents through common chemical route is one of the indirect method to find the constituent phases. Among the direct measurement techniques are quantitative XRD (Clark and Reynolds 1936, Norrish and Taylor 1962, Mandile and Hutton 1995, Hillier 2002), and optical image analysis (Galopin and Henry 1972, Sutherland and Gottlieb 1991, Danti. K.J 1993, Donskoi, Suthers et al. 2007, Lane, Martin et al. 2008), automated image analysis using scanning electron microscopy (SEM) based techniques, such as QemSCAN (Gottlieb, Wilkie et al. 2000, Benvie 2007, Goodall 2008, Lotter 2011) and the Mineral Liberation Analyser (Gu 2003, Fandrich, Gu et al. 2007, Lotter 2011) and in addition to all these process inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis technique was also used to find the mineralogy of trace elements (2001. , Broekaert, Leis et al. 1979, Walsh and Howie 1980, Walsh, Buckley et al. 1981, Boumans 1987, Zhilong, Shuxing et al. 1990). The detailed characterization will help to correlate the characteristics of the iron ore with the design and selection for the beneficiation flow sheet of low grade iron ore fines, which reduces the cost of experimentation and allows to beneficiate a low grade iron ore tailings with a commercial success.

2. Sample collection and characterization methods

Both lump iron ore samples and micro-fine tailing samples are collected from mineral dressing unit and tailing ponds of Donimalai iron ore mines which are subjected to mineralogical, geochemical and liberation analysis. Samples of both lump iron ore and iron ore tailings are grinded and the grain size is reduced is less than 100µm and subjected to X-ray powder diffraction (XRD) analysis for their mineralogical composition. X-ray powder diffraction analysis is carried out using Philips analytical X-ray B.V diffractometer with Fe filtered Cobalt Kα at the scanning rate of 0.02°/Sec at 35kV and 25mA to identify Fe, Al and Si bearing phases and Bruker D8 Advanced X-ray Diffractometer with Ni filtered Copper Kα at the scanning rate of 0.02°/Sec at 35kV and 25mA to identify Fe, Al and Si bearing phases. Microscopic and chemical analysis of Fe, Al and Si-bearing minerals of both lump ore and iron ore tailings are investigated by scanning electron microscope of Hitachi – S3400N at an operating voltage of 15kV. High-resolution imaging was done with the magnification ranging from 200X to 5000X. Elemental mapping and mineral composition of the thin polished samples and powder samples are found out using Horiba EMAX energy EX-400 energy dispersive spectroscopy. The ore samples and the slime samples are grinded and made into less than 150ìm and samples of 25gms weight are taken in Porcelain crucibles. The crucibles are kept inside the air oven at 110±10ºC for 3 hours in order to remove the moisture and the sample are weighed again in order to determine the moisture content in the sample. The moisture removed samples were subjected to elemental analysis using potassium dichromate test and inductive coupled plasma-atomic emission spectroscopy. The main doctrine behind the potassium di chromate test is that ore should be decomposed of hot hydrochloric acid and iron is reduced to its divalent state with stannous chloride. The excess of reductant (i.e. stannous chloride) is removed by oxidation with mercuric chloride. Ferrous iron is determined by titration with standard potassium dichromate solution, employing sodium diphenylamine sulphonate as indicator. The remaining trace elements are analysed using Teledyne Leeman Labs sequential high dispersion inductive coupled plasma- atomic emission spectroscopy (ICP-AES) equipped at National Mineral Development Corporation Ltd., Donimalai. This is PMT based instrument, that is, it uses photo multiplier tube (PMT) as a detector. Silicon is measured at a wavelength of 288.158nm, aluminium at 308.215nm and phosphorous at 214.910nm and the results are matched with NML-Jamshedpur (National Metallurgical Laboratory) standards. Loss on ignition for the iron ore and slime samples are obtained by heating and weighing 1gm of sample inside muffle furnace at 1000 ºC ±10 for 1 hour.

3. Mineralogy

X-ray powder diffraction analysis of both lump ore samples and tailing samples (Fig.1) shows that there is a presence of iron (Fe) bearing minerals and several gangue minerals (Tables 1 and 2) from Donimalai iron ore mines. The main Fe bearing mineral hematite (Fe_2O_3) is identified in the X-ray diffraction pattern by its characteristic peak at 2.6995 Å in Fe filtered Co-Ká run and 2.70300 Å in Ni filtered Cu-Kα, it is confirmed by the PDF numbers 890596 and 24-0072. The gangue minerals such as kaolinite ($Al_2Si_2O_5$ $(OH)_4$) and gibbsite (Al $(OH)_3$) are also identified. From the X-ray diffraction of both lump ore samples and tailings samples we come to know the Fe bearing phase and the gangue minerals do not undergo any phase transition but the quantity of the gangue phases are increased in the tailing samples as compared to the lump ore sample.

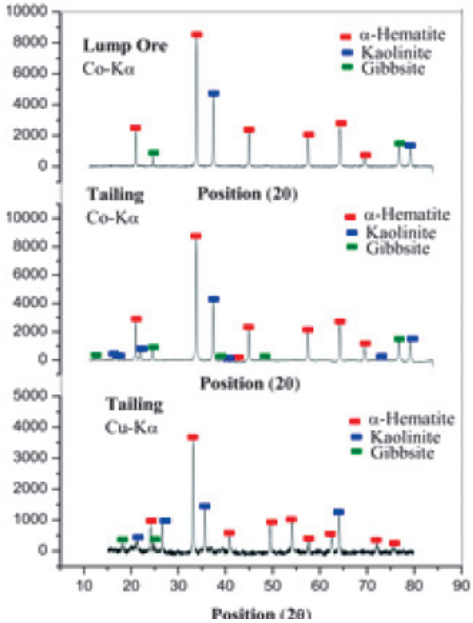


Fig.1 X-ray powder diffraction analysis of (a) lump ore (cobalt Ká) (b) tailing (cobalt Ká) (c) tailing (copper Ká)

4. Major oxides

The quantitative analysis of lump ore and iron ore tailings samples are done by potassium Dichromate test and ICP-MS. In lump ore, the major iron-bearing phase Fe_2O_3 is found to be having a Fe content of 65.36% and the alumina is around 1.28% which is considered to be a permissible limit for blast furnace operation. In the iron ore, tailings the major iron-bearing phase Fe_2O_3 shows Fe content around 49.60% and the alumina (Al_2O_3) is around 8.99% which is above the permissible limit with respect to Indian standards so that it cannot be directly fed into a blast furnace for operation. According to (Murthy and Chattterjee 1995) there is some presence of goethite phase. In order to identify that loss on ignition (LOI) has been carried out for a set of samples. The loss on ignition is found to be of 2.75 to 4.75 which is considered to be the inherent moisture content available. From the loss on ignition test it is clear that the complete recovery of the iron phase will lead to 2.75 to 4.75 per cent addition to the assay.

TABLE 1 XRD DATA OF LUMP ORE FROM DONIMALAI IRON ORE

Sample	Fe filtered Co Kα		Ni filtered Cuká
	Position (2θ)	Mineral identification	
	28.14	α-Hematite	
	31.1	Gibbsite	
	38.84	α-Hematite	
	41.66	Kaolinite	
	47.66	α-Hematite	
	58.18	α-Hematite	
	63.84	α-Hematite	
	68.16	α-Hematite	
Lump ore	74.14	Gibbsite	Not performed
	76.1	Kaolinite	
	28.14	α-Hematite	
	31.1	Gibbsite	
	38.84	α-Hematite	
	41.66	Kaolinite	
	47.66	α-Hematite	
	58.18	α-Hematite	
	63.84	α-Hematite	
	68.16	α-Hematite	

TABLE 2 XRD DATA OF IRON ORE TAILINGS FROM DONIMALAI IRON ORE DEPOSITS

Sample	Fe filtered Co Kα		Ni filtered Cu kα	
	Position (2θ)	Mineral identification	Position (2θ)	Mineral identification
	23.56	Gibbsite	18.251	Gibbsite
	24.273	Kaolinite	21.2723	Kaolinite
	24.79	Kaolinite	24.089	α-Hematite
	28.1386	α-Hematite	24.8377	Gibbsite
	28.989	Kaolinite	26.6152	Kaolinite
	31.056	Gibbsite	33.2386	α-Hematite
	38.7290	α-Hematite	35.6255	α-Hematite
Tailings	41.6556	Kaolinite	36.8596	Kaolinite
	43.02	Gibbsite	40.8832	α-Hematite
	44.16	Kaolinite	49.5201	α-Hematite
	45.05	Kaolinite	54.161	α-Hematite
	46.002	α-Hematite	57.6536	α-Hematite
	47.867	α-Hematite	62.5477	α-Hematite
	51.016	Gibbsite	64.0148	Kaolinite
	53.72	Kaolinite	72.0315	α-Hematite
	58.1693	hematite	75.6329	α-Hematite
	58.97	α-Hematite		
	63.7549	α-Hematite		
	68.078	α-Hematite		
	71.13	kaolinite		
	74.240	Gibbsite		
	76.011	Kaolinite		

5. Size and density analysis

Wet particle size analysis of the tailing sample is conducted by Microtrac S3500 laser diffraction particle size analyser which uses tri laser system with a wavelength of 780nm, has a capability to analyse the particles ranging from 0.01 to 999µm. The microtrac S3500 shows the particles are in the size range of 136µm.

The samples are collected and separated with respect to size by the help of vibratory GEOSYN sieve shaker and standard sieve plates. The measured size distribution is given in the Table 3 and Fig.2. The major amount of slime particles lies in the size range of 180 to 100µm. The liberated fine tailing samples are subjected to density analysis using Accupyc II 1340 helium pycnometer with helium as a testing gas and the density distribution is shown in Table 4. From the density

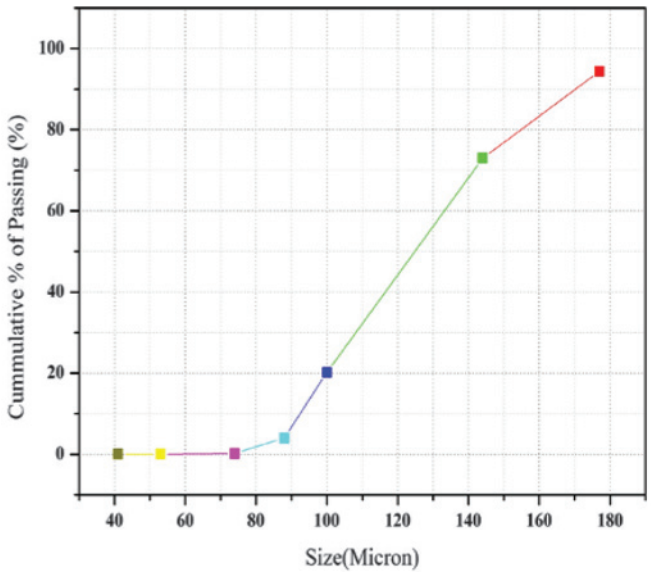


Fig.2 Size distribution of iron ore tailings

TABLE 3 SIZE DISTRIBUTION DATA OF IRON ORE TAILINGS

Size (microns)	Percentage of passing (%)
177	94.3846
144	72.9579
100	2.2022
88	4.0482
74	0.22097
53	0.0569
41	0.0341

TABLE 4 DENSITY OF EACH GRADE AS MEASURED BY HELIUM PYCNOMETRY

Size in µm	Density (g/cm³)
–180+100	4.2003
–100+50	4.0711
–50	3.9731
Composite	4.0815

TABLE 5 SIZE AND CHEMICAL COMPOSITION OF DONIMALAI SLIME

Size in µm	Weight %	Fe%	Al_2O_3	SiO_2
–150 + 50	54.46	62.01	2.83	5.71
–50+20	24.0	56.93	5.32	8.73
–20	21.54	50.02	9.31	13.62
Composite	100	56.32	5.82	9.35

analysis (Table 4) and chemical composition analysis (Table 5) of Donimalai shows that coarser particles are rich in iron bearing elements and the finer fractions shows less Fe (iron) content. The density measurement shows the coarser particles show a maximum presence of hematite phase with less gangue content, whereas the finer fractions are rich in gangue minerals.

6. Microscopic examination

Micro morphological and mineralogical investingation of the iron ore tailings are carried out using scanning electron microscope with EDS attachment. The microscopic examination with spot analysis on the tailings ranging from 180µm to 20µm, shows the presence of hematite and other iron bearing phases present in the sample. The iron bearing phases such as hematite and goethite can be easily identified whereas the magnetite is very difficult to identify due to its similar physical resemblance with hematite. Whereas the silica can be easily identified from other phases. The silica and alumina are present in the form of kaolinite, either on the surface of the grains or between the intergranular spaces. From the EDS analysis (Table 6) it is clear that the finer fraction contains more amount of gangue whereas the coarser fraction has lesser amount.

7. Beneficiation of low-grade tailings

Based on the physical and chemical characteristics of the iron ore tailings obtained from donimalai iron ore deposits (DIOD), the process for beneficiation of the low grade iron ores were selected. The process which was selected should be economically feasible. For example the tailings can be processed by froth flotation or magnetic separation. But it may increase the cost of production and not even required for this sample, particles will go down through spigot and the finer fraction will move upwards through vortex finder.

The process selected for beneficiation is hydrocyclone. The hydrocyclone is static, continuous particle size separation device which uses the centrifugal force i.e. the particles are dispersed into the fluid and enters the cyclone tangential and spirals downwards during the process, the coarser. The test is carried out with Mozley C700 test rig with cyclone size of 2 inch and sump capacity of 40 litres and pump delivery of 3.5m^3/h. The experimentation is carried out with various spigot (3.2,4.5,6.4mm) and vortex (14.0,11.0,8.0mm) diameter with the operating pressure of 0.2 bar to 1.2 bar and the slurry concentration is maintained from 10-30%. It is

TABLE 6 EDS ANALYSIS OF COARSER AND FINER PARTICLES

Elemental composition (%)	Coarser slime particles	Finer slime particles
O	36.81	43.19
Al	5.49	8.92
Si	5.93	11.08
Fe	51.78	36.81

TABLE 7 CHEMICAL ANALYSIS OF HYDRO CYCLONE TEST RESULTS OF DIOD IRON ORE TAILINGS

Stream	Solid recovery	Fe	Sio_2	Al_2O_3
Feed to cyclone	100	56.32	9.35	5.82
Vortex overflow	68	50.6	7.85	4.6
Spigot underflow	32	62.04	1.78	2.12

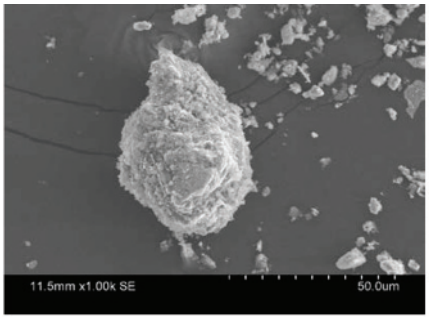
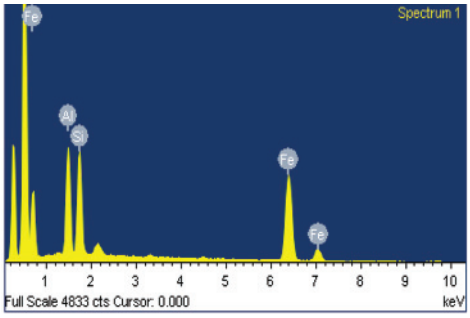


Fig.3a



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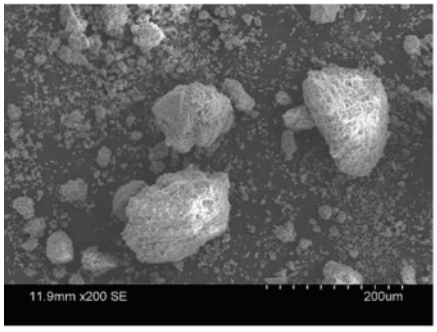

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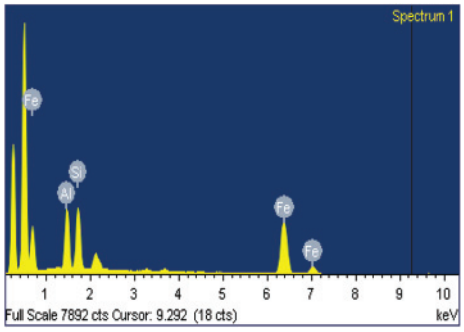

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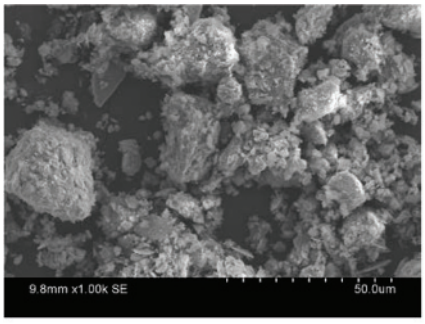

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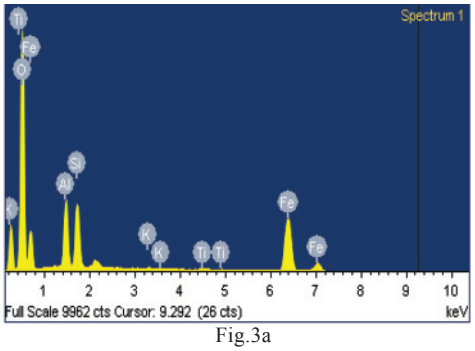

Fig.3a

Figs.3(a,b), 4(a,b) and 5(a,b), SEM photomicrograph and EDS analysis of tailings of 100µm, 200 µm and composite samples

observed that the optimum separation is done at 20% solid with 14mm vortex dia and 4.5mm spigot with operating pressure of 0.6bar. The samples are collected from underflow and overflow and subjected to chemical analysis. The summary of experimental results were given in Table 7.

8. Conclusion

The research work carried out in this paper shows the possibility of upgrading the low grade iron ore tailings obtained from the deposits of Donimalai iron ore deposits. So the upgraded or beneficiated iron ore tailings are used as a feed stock material for blast furnace operation. The tailings which are obtained from the tailing ponds are found to be 56.32% Fe, 9.35% SiO_2, 5.82% Al_2O_3, 0.057% P and 2.60% loss on ignition (LOI) and has a particle size less than 180 µm; so current research work has been carried out for beneficiating the low grade iron ore. So mineralogical, physical and chemical characteristics followed by detailed liberation study are found out for the tailing samples. From the liberation study we come to know about that coarser particles in the tailings are rich in high iron content, whereas the finer fractions have high gangue minerals. So a pilot plant study is conducted with a standard mozley c700, 2 -inch hydrocyclone in order to separate the finer fractions from the tailings. The samples obtained after beneficiation is weighed and analysed, the final product has 62.04% Fe, 1.78 % SiO_2 and 2.12 Al_2O_3 with a recovery rate of 32%. From the work we come to the tailings from the Donimalai deposits can be recovered and can be used as a feedstock material for blast furnace operation after agglomeration.

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