

## ABSTRACT

### ECO-FRIENDLY PROCESS FOR BENEFICIATING IRON ORE SLIMES/FINES

Disclosed is an eco-friendly process for beneficiating iron ore slimes/fines wherein the pH of the to-be beneficiated iron ore is utilized as a criterion for achieving acceptable grades of iron ore. The eco-friendly process eliminates the need of adding external reagent at any stage of the process.

*To be published with Figure 2*

**WE CLAIM:**

- 1) An eco-friendly process for beneficiating iron ore slimes or fines, characterized in obviating addition of external reagents during the process, comprising: adjusting pH of the iron ore slimes or fines to be beneficiated towards high alkalinity prior to processing.
- 2) The eco-friendly process of claim 1, wherein the process obviates addition of external reagents selected from a group comprising dispersants, flocculants and collectors.
- 3) The eco-friendly process of claim 1, wherein the pH of the iron ore slimes or fines to be beneficiated is adjusted ranging from about 10.5 to 12, and preferably at 11.5.
- 4) The eco-friendly process of claim 1, wherein the processing of iron ore comprises batch process, continuous process or a combination thereof for iron ore slimes or fines beneficiation.
- 5) The eco-friendly process of claim 4, wherein the batch process comprises:  
preparing iron ore slime or fines slurry of desired pulp density;  
re-adjusting pH of the iron ore slime or fines slurry for any observable change;  
ultra-sonicating pH adjusted iron ore slime or fines slurry;  
allowing further stirring and later settling to obtain supernatant; and  
repeating successively step of decanting the supernatant and collecting settled portions for predetermined number of times.
- 6) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry of pulp density 5% to 40%, and preferably 10% is prepared.
- 7) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry is readjusted to pH ranging from about 10.5 to 12, preferably 11.5 if any change in previously adjusted pH is observed.

8) The eco-friendly process of claim 5, wherein the pH adjusted iron ore fines slurry is agitated and intermixed in any manner known in the art, and preferably by ultrasonication and stirring.

9) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry is allowed to settle firstly for approximately 1-60 minutes, based upon dimensions of vessel containing the slime or fine.

10) The eco-friendly process of claim 5, wherein the step of decanting, and collecting the settled portions is repeated for the predetermined number of times ranging between 8 to 10.

11) The eco-friendly process of claim 4, wherein the *continuous process is carried in* Floatex density separator or hydrocyclone or spiral classifier.

12) The eco-friendly process of claim 1, wherein the process yields no less than 67% iron grade in beneficiated settled portion with an iron recovery ranging 62% to 69%.

13) The eco-friendly process of claim 1, wherein the process reduces aluminum oxide content in the beneficiated settled portion in range of 2.5% - 3.4%.

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## **FIELD OF THE INVENTION**

The present invention relates generally to a process for beneficiation of iron ore slimes/fines, and more particularly to an eco-friendly process for beneficiation, by adjusting pH of iron ore slimes/fines to be beneficiated.

## **BACKGROUND OF THE INVENTION**

Since the demand for high-grade iron ore slimes/fines is increasing day by day and their presence is limited, the mining industry has no other choice but to utilize low-grade iron ore slimes/fines. Moreover, these low grade iron ore slimes/fines and tailings occupy land in the form of tailing ponds that are environmental hazards. In general, iron ore slimes/fines mainly consist of iron bearing minerals such as hematite and goethite as well as gangue minerals like gibbsite and kaolinite. The mining industry is facing challenges in processing the iron ore inundated with alumina that are present in the ore as the gangue minerals, since there are adverse effects associated therewith. Thus, in order to enhance the utility of iron ore fines/slimes generated during mining and processing of the ore, efficient removal of alumina is absolutely imperative.

The increasing demand for high-grade iron ore has now forced the researchers to look at possible options of beneficiating low-grade iron ore slimes and fines. Additionally, tailing ponds lock a large amount of land and pose an environmental hazard. Thus, the sustainability of iron and steel industry depends on a viable solution to this problem. Several research groups are working towards developing different methods such as, selective dispersion-flocculation, flotation, magnetic separation, hydro cyclones and gravitational settling for the beneficiation of slimes/fines.

It has been reported that the presence of gibbsite and/or kaolinite as the major alumina containing minerals in iron ores does dictate the choice of beneficiation flow sheet. Since these particles are extremely fine sized, their nature at the surface is observed quiet complex. These

aspects of particle characteristics have a strong bearing on any technique that depends on surface properties to induce such particle separation. For example, while kaolinite does not interfere with flotation selectivity, gibbsite tends to contaminate the flotation concentrate as it is depressed together with iron oxides and hydroxides during the reverse cationic flotation process (Araujo et al, 2003). These observations are remarkably close to what Pradip and co-workers (Pradip et al, 1993; Pradip, 1994; Pradip, 1997; Das et al, 1992) have reported based on their investigations on the beneficiation of Indian iron ore slimes by flotation and selective flocculation. Gibbsite tends to go with iron oxide because of its surface chemistry and chelating chemistry being very similar to iron oxide minerals. In fact, the separation of iron oxide from gibbsite continues to remain a challenge before mineral engineers.

As mentioned earlier, the researchers resort to various methods to induce separation of gangue minerals from iron ore. However, these methods pose various other irreparable damages, especially environment related, whenever these gangue minerals are discarded as tailings. For example, in selective dispersion-flocculation process, dispersants are used to stabilize the particles in the suspension and flocculants are used to selectively flocculate the desired mineral. Inorganic dispersants like sodium silicate and sodium hexa-metaphosphate (SHMP) were used in the flocculation experiments and it was found that SHMP was better dispersant in systems where clays are involved (Attia, 1977; Krishnan and Iwasaki, 1983). There are polymeric dispersants such as polyethylene Oxide (PEO) (Attia, 1977; Attia and Deason, 1989; Mathur and Moudgil, 1997) and Polyvinyl Pyrrolidone (PVP) (Ravishankar and Pradip, 1988; Pradip et al, 1993) used in flocculation. Flocculants such as polyacrylamide (PAM), polyacrylic acid (PAA) and starches have been used by many researchers (Gururaj et al., 1983; HanumanthaRao and Narasimhan, 1985; Pradip et al., 1993; Weissenborn et al., 1994; Mathur et al., 2000). Adsorption mechanism of polysaccharides on mineral surfaces was also studied (Liu et al., 2000; Laskowski et al., 2007). At lower pH or in the presence of electrolyte bridging flocculation is observed in starch-kaolinite system (Ma and Bruckard, 2010).

Reagents like sodium oleate, fatty acids have been used to float hematite (Shibata and Fuerstenau, 2003; Pascoe and Doherty, 1997; Thella et al., 2010). In reverse cationic flotation, starch is used to depress iron oxide minerals and amines are used as collectors (Thella et al.,

2010; Rocha et al., 2010). Recent efforts to improve iron ore fines grade by gravity separation by utilizing the specific gravity differences between desired and gangue mineral are found in literature (Subrata Roy, 2009; Claude Bazin et al., 2012; Raghukumar et al., 2012).

The impact of adding these reagents to the process of beneficiation is extremely hazardous to environment around, and in wake of growing cognizance of growing green, adopting such reagent-based methods for beneficiation may not be idealistic.

If, conversely a reagentless process can be devised for achieving efficient beneficiation of iron ore slimes/fines, it may constitute a technical advancement of significant merit to the existing arts.

## **SUMMARY**

This summary is provided to introduce a selection of concepts in a simplified form that are further described below in the detailed description. This summary is not intended to identify key features or essential features of the claimed subject matter, nor is it intended to be used as an aid in determining the scope of the claimed subject matter.

In one aspect the present invention provides an eco-friendly process for beneficiating iron ore slimes or fines that is characterized in obviating addition of external reagents during the process. The process herein comprises adjusting pH of the iron ore slimes or fines to be beneficiated towards high alkalinity prior to processing.

In one other aspect of the invention, the processing of iron ore slime or fine may be achieved using a batch or a continuous process.

In one other significant aspect of the invention, the iron ore slime or fines slurry to be beneficiated is adjusted in range of pH 10.5 to 12, and preferably 11.5.

Additional features and advantages of the invention will be set forth in the description which follows, and in part will be obvious from the description, or may be learned by the practice of the invention. These and other features of the present invention will become more fully apparent from the following description, or may be learned by the practice of the invention as set forth hereinafter.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

The foregoing summary, as well as the following detailed description of preferred embodiments, is better understood when read in conjunction with the appended drawings. For the purpose of illustrating the invention, there is shown in the drawings example constructions of the invention; however, the invention is not limited to the specific composite disclosed in the drawings:

Figure 1 is a flow diagram illustrating a batch process for beneficiating iron ore slimes/fines according to an embodiment of the present invention.

Figure 2 is a graphical representation of effect of pH on yield and iron grade in accordance with one other embodiment of the present invention.

Figure 3 is a flow diagram illustrating a semi-batch process for beneficiating iron ore slimes/fines according to an embodiment of the present invention.

Figure 4 a flow diagram illustrating a continuous process for beneficiating iron ore slimes/fines according to an embodiment of the present invention.

#### **DETAILED DESCRIPTION OF THE INVENTION**

Some embodiments of this invention, illustrating all its features, will now be discussed in detail. The words "comprising," "having," "containing," and "including," and other forms thereof, are intended to be equivalent in meaning and be open ended in that an item or items

following any one of these words is not meant to be an exhaustive listing of such item or items, or meant to be limited to only the listed item or items.

It must also be noted that as used herein and in the appended claims, the singular forms "a," "an," and "the" include plural references unless the context clearly dictates otherwise.

The invention is a process for beneficiating iron ore slimes and fines. In preferred embodiments, iron ore slimes and fines are recovered by a separation method that exploits the mineral surface charge and the particle terminal velocity to achieve said beneficiation. Those skilled in the art understand that the terms "beneficiate", "beneficiation", and "beneficiated" refer to an ore enrichment process in which the concentration of the desired mineral and/or metal in the ore increases as the process proceeds. The separation method utilizes pH of the iron ore slimes or fines as a parameter to achieve acceptable grades of iron ore without addition of any external reagents. The reagents here refer to dispersants, flocculants and the collectors.

While practicing the present invention, the natural iron ore sample of particle size smaller than about 400 mesh or of size equivalent to 37 micron is preferably used as an iron ore feed (slurry). With large particle size, an appreciable amount of the gangue minerals may remain locked in the ore sample to be beneficiated. The slime sample used here, assays 60.4% iron (Fe), 7.7% alumina ( $Al_2O_3$ ) and 5.45% Loss on Ignition (LOI). The present iron ore sample is selectively beneficiated from certain gangue minerals, particularly, gibbsite and kaolinite minerals.

The iron ore slurry may be formed in various ways known to the person skilled in the art. The pH of the slurry is then adjusted to a desired pH range of about 10 to 12.5, and preferably 11.5. The desired pH range may be obtained by adding a pH modifier (acid and base), namely and preferably sodium hydroxide and nitric acid. It shall however be understood that pH adjustment to obtain desired pH range may be accomplished using methods known to those skilled in the art.

In accordance with a preferred embodiment, the beneficiation of iron ore slurry is performed in the pH range of about 10.5 to 12, more preferably at a pH of about 11.5. Thereon the slurry is subjected to further processing by any beneficiation processes known to those skilled in the art, such as batch process, semi-batch process and continuous process.

Considering Batch process of beneficiation and referring to Figure 1, schematics of the experimental procedure are illustrated. The slurry (also known as pulp or pulp slurry) of pulp density ranging from about 5 to 40%, and more preferably at about 10% pulp solids is prepared. Non-limiting example of preparing slurry of pulp density 10% may include addition of dry slime sample to 800 ml of distilled water. The aqueous slurry is then adjusted to a preferred pH range-11.5 and ultra-sonicated for 5 minutes at 30% amplitude on Branson sonifier probe (Model: 102C). Many of the agitating and intermixing techniques are known in the art, and ultrasonication shall not be considered as a limiting example of the same. This pH is re-adjusted in range of from about 10.5-12, and preferably 11.5 if any change is observed. It is stirred further for 30 minutes at 100 rpm and for 3 minutes at 40 rpm and allowed to settle for 1 minute (without stirring). After 1 minute settling, the supernatant is decanted and allowed for settling for 60-90 seconds and decanted. The settling time of 1 minute may vary from 1-60 minutes based upon the dimensions of the vessel containing the slime/fine, and hence shall not be construed as limiting the invention in its present embodiment. The similar decantation procedure is performed for 8-10 times and all the settled portions are mixed together, dried and analyzed by wet chemical analysis.

The result of studying the effect of pH during beneficiation process is presented in Figure 2. The pH of the slurry is varied from acidic to basic values of 4 to 12 and experiment is then conducted with natural slime slurry at 10% pulp density and for 1 minute settling time. As shown in Figure 2, the iron grade in settled portion improved with increase in the basicity; at pH of 11.5 recovery of iron grade of more than 67% is achieved.

The subsequent series decantation experiment is performed at pH 11.5. After stirring, the suspension is allowed to settle for 1 minute and supernatant is decanted. The first settled portion is, labeled as F1. The decanted supernatant is again allowed to settle and supernatant is

decanted. The process of settling and decantation is done for several times (about 8-10 times). All the settled portions is mixed together and analyzed. The results are as shown in the Table 1 below. The overall Fe grade of settled portion achieved is 67.7-68% with a Fe recovery of 62-69%. % Al<sub>2</sub>O<sub>3</sub> in the settled fractions is also very low (2.6-3.4%).

TABLE 1: Series decantation result

Sample ID	Yield, %	Fe Grade	Al <sub>2</sub> O <sub>3</sub> , %	LOI, %
F1	22.7	67.4	3.4	3.2
F2	33.3	68	2.6	2.5
F2t	0.83	63.2	4.0	5.5
S	43.1	50.5	11.9	8.5

Sample details

F1-First settled portion (settling time 1 minute)

F2-Mixture of settled portions (2 to 10)

F2t-Supernatant of F2

S-Final suspended portion

The experiment is repeated to check the reproducibility and a comparison thereof is presented in the Table 2.

TABLE 2: Reproducibility of experiment

Experiment	Yield, %	Fe Grade	Al <sub>2</sub> O <sub>3</sub> %	LOI %
Trial 1	61.8	67.9	3.3	2.7
Trial 2	56.8	67.7	2.9	2.8

TABLE 2

Next, the Schematic for semi batch process of beneficiating iron ore fines/slimes is given in the Figure 3. Slime slurry of 20% pulp density is prepared at 11.5 pH. The slurry is kept under stirring during the entire process. 800ml of pH-adjusted water is taken in the process tank. The impeller speed in the process tank is kept very low (about 5-10 rpm) and the pH adjusted slurry is supplied to the process tank near the impeller blade by drawing through a pipe. No pump is used to draw the slurry and height difference is utilized to assure the flow of the slurry. The supernatant from the process tank is simultaneously siphoned off from just below the surface of the liquid. The liquid level in the process tank is maintained constant by adjusting the inlet and outlet flow rates (The slurry flow rate into the process tank was equal to the siphoning flow rate). Once the slime slurry feeding is done, pH adjusted water was supplied to the process tank *to maintain the liquid level. After supplying pH adjusted water for some time, experiment is stopped and the samples from process tank and gangue collection tank are dried and analyzed.*

This experiment is done on slime at 20% pulp density in the feed slurry tank. The pH in the system is maintained at 11.5 through the end of the experiment. The left over in the feed tank is labeled as F1. The settled portion in the process tank is labeled as F2. The supernatant is collected in two beakers, dried, and analyzed separately. The settled portion in the process tank comprises of 67.5% Fe and 3% Al<sub>2</sub>O<sub>3</sub> grade with a Fe recovery of 42.8%. The supernatants collected are analyzed as 54-55% Fe grade with Al<sub>2</sub>O<sub>3</sub> content of 9% (shown in Table 3 below). Most of the iron minerals can be recovered in the concentrate by increasing the number of process tanks.

TABLE 3: Semi batch process result

Sample ID	Yield, %	Fe Grade	Al <sub>2</sub> O <sub>3</sub> , %	LOI, %
F1	1.2	68.3	3.0	2.7
F2	38.4	67.5	3.0	2.7
S1	47.5	54.9	9.0	6.4
S2	12.9	54.3	9.0	6.9

TABLE 3

### Sample details

F1-Settled portion in the slurry tank

F2-Concentrate in the process tank

S1 & S2-Supernatant collected

Referring now to Figure 4, a schematic diagram of the continuous process flow for beneficiating iron ore slimes/fines is presented. The iron ore sample (as a feed material) is studied for different and detailed characterization studies. The average size particle of the iron ore slime is kept below 37 microns. The pH of the iron ore slime/fine is adjusted ranging from 10.5 to 12, and more preferably at 11.5. Now, the feed is treated in well-known Floatex Density Separator (FDS) wherein the gangue minerals are separated on the principle of hindered settling and fluidization. Apart from FDS, the feed may alternatively be treated in Hydrocyclone/Spiral Classifier. Upon separation, the gangue rich slurry is de-watered and disposed for reclamation, while the high-grade iron concentrate is de-watered, dried and ultimately pelletized to obtain high iron grade pellets having reduced alumina and gangue content.

The present invention accomplishes the iron ore beneficiation without requiring addition of any external reagent-dispersant, flocculent or collector at any stage of the process, which eventually can reduce the burden of environment bemiring that has off late surfaced as one of the most formidable threat to iron ore processing industry.

It is, therefore, a general object of this invention to provide an eco-friendly method of processing alumina rich iron ore fines or slimes by a process not attainable heretofore.

It is a further object of this invention to provide process that accomplishes the beneficiation by exploiting the surface chemistry and chelating chemistry of the particles and accordingly adjusting the slime pH to attain high grade concentrate assaying >67% Fe and <3%  $\text{Al}_2\text{O}_3$ .

Those skilled in the art will appreciate that there may be many variations and modifications of the process and conditions described herein which are within the scope of the present invention.

The foregoing description of specific embodiments of the present invention by way of examples has been presented for purposes of illustration and description. They are not intended to be exhaustive or to limit the invention to the precise forms disclosed, and obviously many modifications and variations are possible in light of the above teaching. The embodiments were chosen and described in order to best explain the principles of the invention and its practical application, to thereby enable others skilled in the art to best utilize the invention and various embodiments with various modifications as are suited to the particular use contemplated. It is intended that the scope of the invention be defined by the claims appended hereto and their equivalents. The listing of steps within method claims do not imply any particular order to performing the steps, unless explicitly stated in the claim.

**WE CLAIM:**

- 1) An eco-friendly process for beneficiating iron ore slimes or fines, characterized in obviating addition of external reagents during the process, comprising: adjusting pH of the iron ore slimes or fines to be beneficiated towards high alkalinity prior to processing.
- 2) The eco-friendly process of claim 1, wherein the process obviates addition of external reagents selected from a group comprising dispersants, flocculants and collectors.
- 3) The eco-friendly process of claim 1, wherein the pH of the iron ore slimes or fines to be beneficiated is adjusted ranging from about 10.5 to 12, and preferably at 11.5.
- 4) The eco-friendly process of claim 1, wherein the processing of iron ore comprises batch process, continuous process or a combination thereof for iron ore slimes or fines beneficiation.
- 5) The eco-friendly process of claim 4, wherein the batch process comprises:  
preparing iron ore slime or fines slurry of desired pulp density;  
re-adjusting pH of the iron ore slime or fines slurry for any observable change;  
ultra-sonicating pH adjusted iron ore slime or fines slurry;  
allowing further stirring and later settling to obtain supernatant; and  
repeating successively step of decanting the supernatant and collecting settled portions for predetermined number of times.
- 6) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry of pulp density 5% to 40%, and preferably 10% is prepared.
- 7) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry is readjusted to pH ranging from about 10.5 to 12, preferably 11.5 if any change in previously adjusted pH is observed.

8) The eco-friendly process of claim 5, wherein the pH adjusted iron ore fines slurry is agitated and intermixed in any manner known in the art, and preferably by ultrasonication and stirring.

9) The eco-friendly process of claim 5, wherein the iron ore slime or fines slurry is allowed to settle firstly for approximately 1-60 minutes, based upon dimensions of vessel containing the slime or fine.

10) The eco-friendly process of claim 5, wherein the step of decanting, and collecting the settled portions is repeated for the predetermined number of times ranging between 8 to 10.

11) The eco-friendly process of claim 4, wherein the *continuous process is carried in* Floatex density separator or hydrocyclone or spiral classifier.

12) The eco-friendly process of claim 1, wherein the process yields no less than 67% iron grade in beneficiated settled portion with an iron recovery ranging 62% to 69%.

13) The eco-friendly process of claim 1, wherein the process reduces aluminum oxide content in the beneficiated settled portion in range of 2.5% - 3.4%.

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