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ORE BENEFICIATION

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This invention relates as indicated to the art of ore beneficiation and more specifically to a process for separating iron-oxygen compounds from iron bearing ores by flotation.

Iron ores, immediately after they are mined, contain excess quantities of siliceous gangue which make direct transportation and smelting of the ores economically impractical. For this reason, the ores are generally purified by such processes as flotation. Many ore flotation processes have been proposed in the past, both to float the iron constituent of the ore from the gangue and to float the gangue from the iron. These processes however, have been very inefficient in concentrating ores containing high percentages of slimes. Generally in order to produce acceptably pure products by these processes it has been necessary to remove the slimes from the ore before it is floated. The desliming operations considerably increase the cost of the finished ore both because the desliming operations themselves require time and money and because the iron constituents of the slimes are lost.

It is, therefore, the principal object of this invention to provide a process of ore flotation whereby substantially all of the iron content of the ore will be collected with a minimum quantity of gangue. It is a more particular object of this invention to provide a process of iron ore flotation wherein desliming of the ore is not necessary.

Other objects and advantages of the present invention will become apparent as the following description proceeds.

To the accomplishment of the foregoing and related ends, said invention, then, comprises the features hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative, however, of but a few of the various ways in which the principle of the invention may be employed.

Broadly stated our invention comprises a process for the recovery of iron-oxygen compounds from iron bearing ores which comprises grinding said ore to flotation size in the presence of 1 to 5 pounds of a carboxylic acid per ton of solids and 1 to 20 pounds of an oil per ton of solids, and subjecting the entire mass which has thus been ground to froth flotation.

We have found that by grinding iron ores in the presence of a carboxylic acid collector and a hydrocarbon oil, substantially all of the iron constituents of the ore may be collected by flotation. Iron ore which has been ground without reagents has a red color which is characteristic of the dispersed fine sizes of hematite. It is recognized that known flotation processes do not produce satisfactory separation of iron from gangue materials when these fine red colored sizes of iron material are present. The fine sizes of iron materials generally produce large quantities of non-selective froths containing substantial quantities of gangue materials.

When the ore is ground according to our invention in the presence of a carboxylic acid collector and fuel oil, it is found that these fine sizes of iron are heavily flocculated so that they gather in heavy black masses, while the fine sizes of gangue are well dispersed in the ore. In the subsequent flotation of the ground ore, these flocculated masses of fine iron particles are carried to the surface in the froth, and the dispersed gangue particles remain in the tailing. The exact reason for this flocculation of the fine iron particles is not completely understood at present. However, when the ore is ground in the presence of the reagents the fine sizes of iron are floated to the surface with the remainder of the heavy iron concentrates leaving the gangue particles in the tailing. For this reason, it is not necessary to remove the fine particles from ore treated according to our invention. The fine iron particles are collected in the concentrated iron froth, and the fine gangue particles remain in the tailings.

The carboxylic acid collector used may be any one of a number of crude or refined fatty or resin acids. Crude fatty acids such as tall oil and acidulated cottonseed foots have been found to be good collectors to employ in the process of our invention. The amount of carboxylic acid collector used in our process should exceed 1 lb. per ton of the ore and we have found on all the ores tested that it is not necessary to use an amount exceeding 5 lbs. per ton of ore.

One of the oils most economical and easily obtainable for use in the process of our invention is that referred to by the trade as No. 2 fuel oil. This oil is a petroleum fraction having a boiling point of between 450 and 670° F. and a flash point of 100° F. The amounts of fuel oil used in the process should be within the range of 1 to 20 lbs. of oil per ton of solids, and in most cases, we have found that 7 to 10 lbs. of oil per ton of solids is most desirable.

As indicated above the broad embodiment of our invention comprises grinding the ore in the presence of the reagents and subsequently floating the iron constituents thereof from the gangue in a bath of water. On some ores we have found it desirable to preliminarily comminute the ore before the grinding with the reagents and in some cases we have found it desirable to grind the ore in the presence of the reagents, then float the ore collecting the iron constituents thereof, and subsequently fine grinding the ore in the presence of more reagents and subjecting the fine ground material to froth flotation.

Concentration of iron ore by prior art processes has necessitated grinding of the ore to at least approximately 65 mesh. However, we have found that iron particles as large as 20 mesh may be floated by our process. The best products are obtained when the ore is ground to such an extent that the iron is liberated from the gangue. Grinding to 100 mesh is generally sufficient for the purpose. Of course, the degree of grinding necessary to obtain the best product varies with the type of ore being purified. For this reason no specific time limits for grinding can be stated. The required grinding varies for different ores.

Our process can be varied to a considerable degree within the limits of our invention. It is essential that the ore be ground in the presence of the carboxylic acid collector and an oil, but other reagents may be added to the ore immediately prior to flotation and any of the well-known cleaning and finishing operations may be employed after the flotation. We have found it desirable to add to the ground pulp just prior to flotation a small quantity of sodium silicate or Orzan, AH-3.

The following examples are included herein to more particularly illustrate specific methods of practicing our invention on given ores and to illustrate its advantages over prior art processes.

EXAMPLE 1

A sample of Mesabi Range rock rejects was crushed to −10 mesh and the −10 mesh material ground in a laboratory rod mill to liberation size. In the grinding operation the sample was ground for 15 minutes and then the reagent addition of the fuel oil and Aliphat 44B were made to the pulp in the rod mill. The pulp was then ground an additional five minutes with the reagents. The Aliphat 44B is a distilled tall oil produced by General Mills, Inc. After the sample had been ground in the presence of the collector and fuel oil, it was diluted to 13% solids. To the diluted pulp was added a small quantity of sodium silicate. Subsequent to the addition of the sodium silicate, a rougher flotation concentrate was skimmed from the flotation cell. The rougher flotation concentrate was then subjected to three cleaning operations to produce a concentrate of an acceptable grade. The test procedure and results obtained are presented in the following table.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds Per Ton		
	Time, Minutes	Percent Solids	pH	Aliphat 44B	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	15	67	Natural			
Rod Mill Grind	5	67	do	2.04	7.05	
Rougher Float	2	13	do			0.50
1—Cleaner	2		do			0.50
2—Cleaner	2		do			0.25
3—Cleaner	2		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		36.97			
Heads (Calculated)	100.00			36.69	100.00
Rougher Tailing	27.10	5.43		1.47	4.01
1 Cleaner Tailing	11.13	11.25		1.25	3.41
2 Cleaner Tailing	5.33	21.53		1.15	3.13
3 Cleaner Tailing	3.09	32.37		1.00	2.73
Concentrate	53.35	59.65	8.26	31.82	86.72

EXAMPLE 2

A similar sample of Mesabi Range rock rejects was concentrated in this example. The carboxylic acid collector used was Liqro, a crude tall oil product of the West Virginia Pulp and Paper Company. The procedure followed in this example was the same as that followed in Example 1 except that in the present test the reagents were added to the sample at the beginning of the grinding period and the pulp was diluted to 26% solids for flotation. The following table contains the conditions and results of this test.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds per Ton		
	Time, Minutes	Percent Solids	pH	Liqro	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	20	67	Natural	2.34	8.22	
Rougher Float	2½	26	do			0.50
1—Cleaner	2½		do			0.50
2—Cleaner	2½		do			0.25
3—Cleaner	2½		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		36.97			
Heads (Calculated)	100.00			36.47	100.00
Rougher Tailing	28.08	6.47		1.81	4.96
1 Cleaner Tailing	11.31	13.35		1.51	4.14
2 Cleaner Tailing	5.78	24.27		1.40	3.84
3 Cleaner Tailing	3.26	34.48		1.12	3.07
Concentrate	51.57	59.40	9.22	30.63	83.99

EXAMPLE 3

The sample of material used for this test is known as a specular hematite and was obtained from the Groveland formation. In this example, the collector and fuel oil were added to the sample at the beginning of the grinding period. The collector used was acidulated cottonseed foots produced by The Southern Cotton Oil Company. In this test no sodium silicate was used.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds Per Ton	
	Time, Minutes	Percent Solids	pH	Acidulated Cottonseed Foots	No. 2 Fuel Oil
Rod Mill Grind	15	67	Natural	2.20	7.68
Rougher Float	1½	26	do		
1—Cleaner	1½		do		
2—Cleaner	1½		do		
3—Cleaner	1½		do		

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		34.97			
Heads (Calculated)	100.00			35.05	100.00
Rougher Tailing	32.52	5.88		1.91	5.45
1 Cleaner Tailing	9.09	12.65		1.15	3.28
2 Cleaner Tailing	4.10	21.06		0.86	2.45
3 Cleaner Tailing	2.26	28.49		0.64	1.83
Concentrate	52.03	58.60	9.38	30.49	86.99

EXAMPLE 4

The sample used for this test was a drill core sample from the formation known as Felch Mountain. The crushed −10 mesh material was ground in a laboratory rod mill for 15 minutes. The collector, Aliphat 44E, a product of General Mills, Inc., and fuel oil additions were then made to the pulp in the rod mill and the sample ground with the reagents for an additional five minutes. The ground sample which had been conditioned with the above-mentioned reagents during the last stages of the grind was pulped with water in a laboratory flotation cell and a sodium silicate addition made to the pulp. A rougher concentrate was then removed by skimming the froth from the cell. In like manner, the rougher concentrate was cleaned three times by repulping with tap water. Sodium silicate additions were made to the first and second cleaner circuits.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds Per Ton		
	Time, Minutes	Percent Solids	pH	Aliphat 44E	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	15	67	Natural			
Rod Mill Grind	5	67	do	1.63	7.05	
Rougher Float	2½	26	do			0.50
1—Cleaner	2½		do			0.50
2—Cleaner	2½		do			0.25
3—Cleaner	2½		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		32.72			
Heads (Calculated)	100.00			32.91	100.00
Rougher Tailing	31.44	4.62		1.45	4.41
1 Cleaner Tailing	12.00	10.86		1.30	3.95
2 Cleaner Tailing	5.99	21.71		1.30	3.95
3 Cleaner Tailing	3.40	31.42		1.07	3.25
Concentrate	47.17	58.91	6.20	27.79	84.44

Analysis of calcined flotation concentrate

Product	Fe	SiO_2	CaO	MgO	Mn	Al_2O_3	P	S
Concentrate	60.70	6.70	2.66	2.04	0.50	0.12	0.050	0.03

EXAMPLE 5

The invention is further described in this example where the process has been applied to various size fractions of Mesabi Range ores which are too refractory to be concentrated effectively by the conventional gravity methods which are now used. For example the 1½ inch natural size particles of an ore identified as Halobe when crushed to ½" and concentrated by the conventional heavy media method produced a concentrate which assayed 54.00 percent iron and 22.73 percent silica. However, when a sample of the 1½ inch natural size particles of the ore was crushed and ground to flotation size and then concentrated by the method shown in this invention, a concentrate of high grade and recovery was obtained. A description of the test conditions, reagents used, and metallurgical results obtained on the 1½ inch Halobe ore is presented in the following tabulation.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds per Ton		
	Time, Minutes	Percent Solids	pH	Liqro	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	20	67	Natural	2.34	8.22	
Rougher Float	2	26	do			0.50
1—Cleaner	2		do			0.50
2—Cleaner	2		do			0.25
3—Cleaner	2		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		38.19			
Heads (Calculated)	100.00			38.94	100.00
Rougher Tailing	23.15	4.46		1.03	2.65
1 Cleaner Tailing	10.49	9.67		1.01	2.59
2 Cleaner Tailing	5.43	15.87		0.86	2.21
3 Cleaner Tailing	3.13	25.05		0.78	2.00
Concentrate	57.80	61.01	8.65	35.26	90.55

EXAMPLE 6

Application of our flotation process to the large reserves of non-magnetic taconite found on the Mesabi Range is shown in Examples 6 and 7. The non-magnetic taconite sample used in these tests was obtained from the property known as West Patrick. In this example, the crushed —10 mesh material was ground in a laboratory rod mill for 20 minutes. The collector, Liqro, which is a crude tall oil, and fuel oil additions were then made to the pulp in the rod mill and the pulp ground an additional five minutes with the reagents. The ground sample was then pulped in a laboratory flotation cell and a sodium silicate addition made to the pulp. The rougher froth was then skimmed from the flotation cell. In like manner, the rougher concentrate was cleaned three times by repulping with tap water. Additional sodium silicate was added to the first and second cleaning circuits.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds per Ton		
	Time, Minutes	Percent Solids	pH	Liqro	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	20	60	Natural			
Rod Mill Grind	5	60	do	1.71	8.96	
Rougher Float	1½	26	do			0.50
1—Cleaner	1½		do			0.25
2—Cleaner	1½		do			0.25
3—Cleaner	1½		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		35.07			
Heads (Calculated)	100.00			35.83	100.00
Rougher Tailing	27.16	2.50		0.68	1.90
1 Cleaner Tailing	11.77	5.25		0.62	1.73
2 Cleaner Tailing	5.05	11.13		0.56	1.56
3 Cleaner Tailing	2.49	20.50		0.51	1.42
Concentrate	53.53	62.50	8.97	33.46	93.39

EXAMPLE 7

The test shown in this example is a duplicate of Example 6 except that slightly decreased quantities of fatty acid and fuel oil were used. The conditions, reagents used, and metallurgical results of the test are presented in the following table.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds per Ton		
	Time, Minutes	Percent Solids	pH	Liqro	No. 2 Fuel Oil	Sodium Silicate
Rod Mill Grind	20	60	Natural			
Rod Mill Grind	5	60	do	1.52	8.86	
Rougher Float	2	26	do			0.50
1—Cleaner	2		do			0.25
2—Cleaner	2		do			0.25
3—Cleaner	2		do			

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (Assayed)		35.07			
Heads (Calculated)	100.00			35.70	100.00
Rougher Tailing	30.83	3.38		1.04	2.91
1 Cleaner Tailing	11.77	9.13		1.07	3.00
2 Cleaner Tailing	5.18	20.00		1.04	2.91
3 Cleaner Tailing	2.89	31.63		0.91	2.55
Concentrate	49.33	64.13	7.09	31.64	88.63

COMPARATIVE EXAMPLE OF PRIOR ART

In order to further illustrate the improved results that have been obtained by the process presented in this invention, a test was conducted using the known conventional petroleum sulfonate process. The test using the petroleum sulfonate procedure was performed on a sample of the same ore which was used in the test described in Example 1. It is well known that in order for the sulfonate process to function satisfactorily the fine sizes of ground ore frequently referred to as slimes must be first removed in order that their adverse effect on the subsequent flotation operation will be eliminated. The procedure and results obtained with the sulfonate process are presented in the following table. In conducting this test the —10 mesh ore was ground for 20 minutes in a rod mill. After grinding the sample was pulped to 20% solids, the fines were dispersed with sodium silicate and subsequently removed after the pulp had settled for three minutes. The desliming operation was repeated a second time to effectively remove a sufficient quantity of the —10 to —15 micron size particles. The deslimed pulp was then conditioned at high solids with American Cyanamid Reagent 899, fuel oil and sulphuric acid. A rougher flotation concentrate was cleaned three times in order that an iron concentrate of an acceptable grade would be produced.

Conditions and reagents

Stage of Process	Conditions			Reagents Pounds Per Ton			
	Time, Minutes	Percent Solids	pH	Reagent 899	Fuel Oil	Sulphuric Acid	Sod. Silicate
Rod Mill Grind	20	67					
Deslime (2 times)	3	20					6.0
Condition	3	50		5.0	3.76	7.0	
Rougher Float	2		4.0				
1—Cleaner	2						
2—Cleaner	2						
3—Cleaner	2						

Metallurgical results

Product	Weight Percent	Assay Percent		Units Fe	Distribution Percent Fe
		Fe	SiO_2		
Heads (assayed)		36.97			
Heads (calculated)	100.00			37.57	100.00
Slime	31.28	31.33		9.80	26.08
Rougher Tailing	10.48	7.55		0.79	2.10
1 Cleaner Tailing	11.04	10.06		1.11	2.95
2 Cleaner Tailing	5.86	22.87		1.34	3.57
3 Cleaner Tailing	5.04	41.90	35.78	2.11	5.62
Concentrate	36.30	61.76	7.33	22.42	59.68
Concentrate and 3 Cleaner Tailing	41.34	59.34	10.79	24.53	65.30

In comparing the results obtained with the new process described in this invention with the results obtained with a known conventional process, it is clearly seen that a considerably higher yield of iron was obtained by effectively concentrating the iron in the slime fraction. Thus the concentrate product obtained by the prior art processes contained only 59.68% of the iron in the original ore. While the concentrate product obtained in Example 1 contained 86.72% of the iron in the original ore, the two products were of approximately the same purity. The product in Example 1 was 59.65% iron and the prior art product was 61.76%.

The process of our invention performs the direct object for which these flotation processes were proposed. Our process produces a product containing substantially all of the iron in the original ore. The product of our process is substantially pure so that undue costs of shipping and smelting a large quantity of siliceous gangue are not incurred. In our process, the slimes, i.e. fine particles in the ground ore need not be removed from the ore before flotation. In the flotation operation, the iron constituents of the slimes are collected in the finished product and the gangue constituents of the slimes are left behind in the tailings.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims or the equivalent of such be employed.

We, therefore, particularly point out and distinctly claim as our invention:

1. A process for the recovery of iron-oxygen compounds from iron bearing ores which comprises grinding said ore to flotation size in the presence of 1 to 5 pounds of a fatty acid material per ton of solids and 1 to 20 pounds of a hydrocarbon oil per ton of solids, and subjecting the entire mass which has thus been ground to froth flotation.

2. The process of claim 1 characterized further in that said ore is ground to such an extent that 95% thereof will pass a 20 mesh screen.

3. A process for the recovery of iron-oxygen compounds from iron bearing ores which comprises preliminarily comminuting the ore, fine grinding that portion of such preliminarily comminuted mass which contains all of the fines including the slime fines to flotation size in the presence of 1 to 5 pounds of a fatty acid per ton of solids and 1 to 20 pounds of a mineral oil per ton of solids, and subjecting the entire mass which has thus been finely ground to froth flotation.

4. The process of claim 3 characterized further in that said ore is comminuted to such an extent that 95% thereof will pass a 20 mesh screen, and the preliminarily comminuted ore which contains all of the fines is finely ground to such an extent that 95% thereof will pass a 100 mesh screen.

5. A process for the recovery of iron-oxygen compounds from iron bearing ores which comprises preliminarily grinding said ore to flotation size in the presence of 1 to 5 pounds of a fatty acid per ton of solids and 1 to 20 pounds of a mineral oil per ton of solids, subjecting the entire mass which has thus been preliminarily ground to froth flotation, and collecting the portion thereof which floats, fine grinding such collected portion in the presence of up to 5 pounds of a fatty acid per ton of solids and up to 20 pounds of a mineral oil per ton of solids, and subjecting the entire mass which has thus been finely ground to froth flotation to recover as well the iron-oxygen compounds contained in the slimes of said ore.

6. The process of claim 5 characterized further in that said ore is preliminarily ground to such an extent that 95% thereof will pass a 20 mesh screen and said collected portion is ground to such an extent that 95% thereof will pass a 100 mesh screen.

7. The process of claim 1 characterized further in that said fatty acid material is a resin acid.

8. The process of claim 1 characterized further in that said fatty acid material is tall oil.

9. The process of claim 1 characterized further in that said fatty acid material is acidulated cottonseed foots.

10. The process of claim 1 characterized further in that said hydrocarbon oil is a mineral oil.

11. The process of claim 1 characterized further in that said hydrocarbon oil is a petroleum oil fraction boiling within the range from 450° to 670° F.

12. A process for the recovery of iron-oxygen compounds from hematite ores which comprises grinding said ore to liberation size in the presence of 1 to 5 pounds of tall oil per ton of ore and 7 to 10 pounds of a petroleum oil boiling within the range of from 450° to 670° F. per ton of ore, subjecting the entire mass which has thus been ground including the slime fines to froth flotation in water, and collecting the iron-bearing froth which floats.

13. A process for the recovery of iron-oxide compounds from iron bearing ores which comprises grinding said ore to flotation size, adding up to 5 pounds of a carboxylic fatty acid and up to 20 pounds of a hydrocarbonaceous oil per ton of solids to the ore simultaneously with the grinding step, flocculating the iron minerals and iron slimes of the ore during the grinding step by said acid and oil additives, and subjecting the entire mass to froth flotation.

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