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Iron Oxide Red Pigment Prepared from Pyrite Cinders

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Abstract: A new technology of recovering iron oxide red pigment from pyrite cinders was developed. The results indicate that the main component of pyrite cinder is Fe, followed by SiO_2, Al_2O_3 and S. By the process of classification, rinsing, fine grinding and reverse flotation, mechanically activated and oxidation roasting, an iron oxide red which accords with the China's national iron oxide red pigment product quality standard can be obtained. The process is relatively simple, low cost and easy-to-production implementation.

Key words: Pyrite cinders, classification, fine rinsing, flotation, iron oxide red pigment

INTRODUCTION

Annually, a large amount of sulfuric acid are produced in china. Pyrite is commonly used as the raw material (Tveit, 2003). The process involves the roasting of pyrite concentrates to generate sulfur dioxide gas followed by its subsequent catalytic oxidation to sulfur trioxide gas prior to the eventual conversion of the latter to sulfuric acid. The roasting process also yields solid wastes known as pyrite cinders, which are composed mainly of iron oxides (Alp *et al*., 2009). Theoretically, around 67% of pyrite in the feed is converted into hematite (Fe_2O_3) during the roasting process. Therefore, large quantities of pyrite cinders as solid wastes are produced as a by-product of industrial sulfuric acid manufacturing operations. The pyrite cinder contains hazardous heavy metals and acids which are potential environmental risks for disposal (He *et al*., 2010). Nowadays, only a small amount of pyrite cinders is recycled as building materials, the rest is dumped as solid wastes, not only occupying much land, but also causing a great waste of iron resources. It also poses a serious threat to the environment, public health and safety due to the release of acids and toxic substances (Tugrul *et al*., 2003).

Although pyrite cinder is industrial waste, it is also a kind of potential resource as it contains Fe_2O_3, Fe_3O_4, SiO_2, CaO, S etc. During the past decade, considerable researches were devoted to the comprehensive utilization of pyrite cinder. At present, several areas for potential use of pyrite cinder are reported, including recycling of the contained metals (Ishimitsu, 1969; Xu, 2009; Li *et al*., 2009; Shang *et al*., 2009; Zou *et al*., 2005), using as brick-making materials (Abdrakhimov *et al*., 2006; Tsakiridis *et al*., 2008), preparing iron-based pigments like iron oxide red and iron black (Yang *et al*., 2006; Zheng and Fu, 2007; Liu *et al*., 2010; Chen and Kong, 2008).

The aim of this work is to develop an economical and environmentally acceptable method to recover iron oxide red pigment from pyrite cinder. In the experiment, the pyrite cinder which contains iron oxide, silica, sulphur and alumina was classified, rinsed, fine grinded, reverse floated, mechanically activated and oxidation roasting orderly to produce iron oxide red pigment. An iron oxide red pigment production which accords with the China's national iron oxide red pigment product quality standard was produced in the process.

MATERIALS AND METHODS

Samples and reagents: The pyrite cinder sample used in this study was taken from Yunfu, Guangdong Province, China. The chemical compositions of raw material were determined by X-ray fluorescence (Fig. 1) and the results were shown in Table 1. It can be observed that the raw sample was appeared to be a ready source of iron with a total iron content of 62.63%. The chemical analysis of the sample was also revealed that this sample contained impurities, which mainly were SiO_2 and Al_2O_3. All reagent used in this study were analytical grade except the Na_2SiO_3, which was industrial grade.

Procedure: Figure 2 is the representative flowsheet of recovery iron oxide red from Pyrite cinder. The raw pyrite cinder was screened at 147 μm and the coarse fraction was dropped. The -147 μm fraction was rinsed to reduce soluble salts and improve flotation pH conditions. After

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the screening and rinsing process, the -147 μm fraction of pyrite cinder became milling and flotation feed. The 1.0 kg portions of the ore sample were milled to achieve a grind of 100% passing 10 μm using a laboratory scale stainless steel rod mill. The milled slurry was transferred to a 3.0L laboratory XFG cell. The air flow rate was kept at 0.1 N m^3 h^{-1} monitored with a flow meter and impeller speed which was set at 1200 rpm. By the process of mechanical activation,concentrate, drying,oxygenize roasting,an iron oxide red pigment product can be obtained. The reagent found to give the best iron recovery is shown in Fig. 2.

X-ray diffraction: XRD measurements were carried out by X-ray diffraction on the diffractometer (Rigaku D/max 2500 PC, Japan) using Cu Kα radiation (λ = 1.54 A, voltage 40 kV, current 20 mA) with time constant 0.5 sec, scanning speed 2 (°)/min.

Scanning electron microscopy: Scanning electron microscopy in combination with EDAX was performed with an Quanta 200 (FEI, Hong Kong).

Characteristic measurements of product: Characteristic of products was measured according to GB1863-2008 (China Industrial Standard). The oil absorption and color of iron oxide red are comparison with the standard sample from Hunan Three-ring Pigment Co., China. The results were the mean values of the two experiments using the same sample.

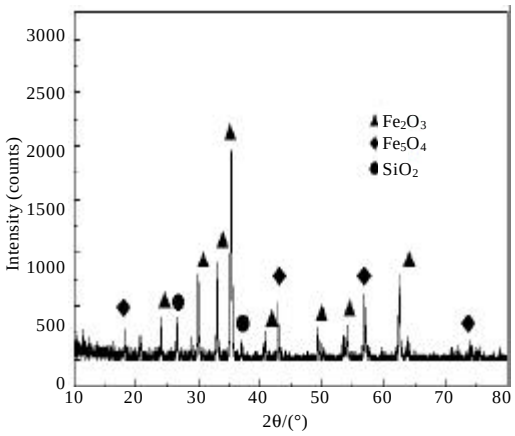

Fig. 1: XRD pattern of pyrite cinder

RESULTS AND DISCUSSION

Screening and rinsing: The screen result shows that the coarser the grain of pyrite cinder is, the lower iron content is. Therefore it would be helpful to improve iron content of raw materials and reduce grinding and flotation costs by sieving to remove coarse fraction. A particle size of 147 μm was selected for the classification optimisation. Due to the presence of soluble salts of $FeSO_4$, $Fe_2(SO_4)_3$, $CuSO_4$, $MgSO_4$ and $CaSO_4$, the pH of pyrite cinder slurry was low (pH = 4 or so), which not only result in serious corrosion to the device, but also interfere with the separation of iron mineral from impurities. It will be helpful to improve the flotation condition by reducing the amount of soluble salts and changing the pH from acidic to alkaline. To achieve this objective, the ores were washed by water. Rinsing water overflow was treated by wastewater treatment system. The results of screening and rinsing process are shown in Table 2.

Flotation: The pulp is easy to flocculation as a result of the high specific surface area of the fine particles as well as the inevitable ions exist in the pulp. The effective and stable dispersion of pulp is the premise and basis of flotation. The sodium hydroxide and sodium silicate are added to the pulp to improve the dispersion state of mineral particles. Starch is used as depressant of hematite and lauryl amine is used as collector of silicate mineral. Based on the optimized of experimental conditions, results of flotation circuit which recovers iron concentrate form pyrite cinder are listed in Table 3. It can be seen from Table 3 that an iron concentrate can be obtained by the process of classification,rinsing,fine grinding and reverse flotation with a recovery of 65.96%.

Table 1: Chemical composition of sample (wt%)

Components	TFe	S	SiO_2	Al_2O_3	CaO	MgO	Mn	Pb	Zn	Cu	FeO
Content (%)	62.63	0.32	4.02	0.34	0.33	0.12	0.07	0.10	0.09	0.008	2.62

Table 2: Result of screening and rinsing process (%)

Particle size interval (μm)	+147	-147+104	-104+74	-74+37	-37
Weight	1.50	3.12	21.28	37.87	36.23
Iron grade	48.88	62.84	62.89	63.07	63.12
Silica grade	18.06	7.28	3.48	3.87	3.89
S grade	0.37	0.34	0.12	0.12	0.12
Iron monomer dissociation rate	57.5	79.4	83.5	95.4	91.5

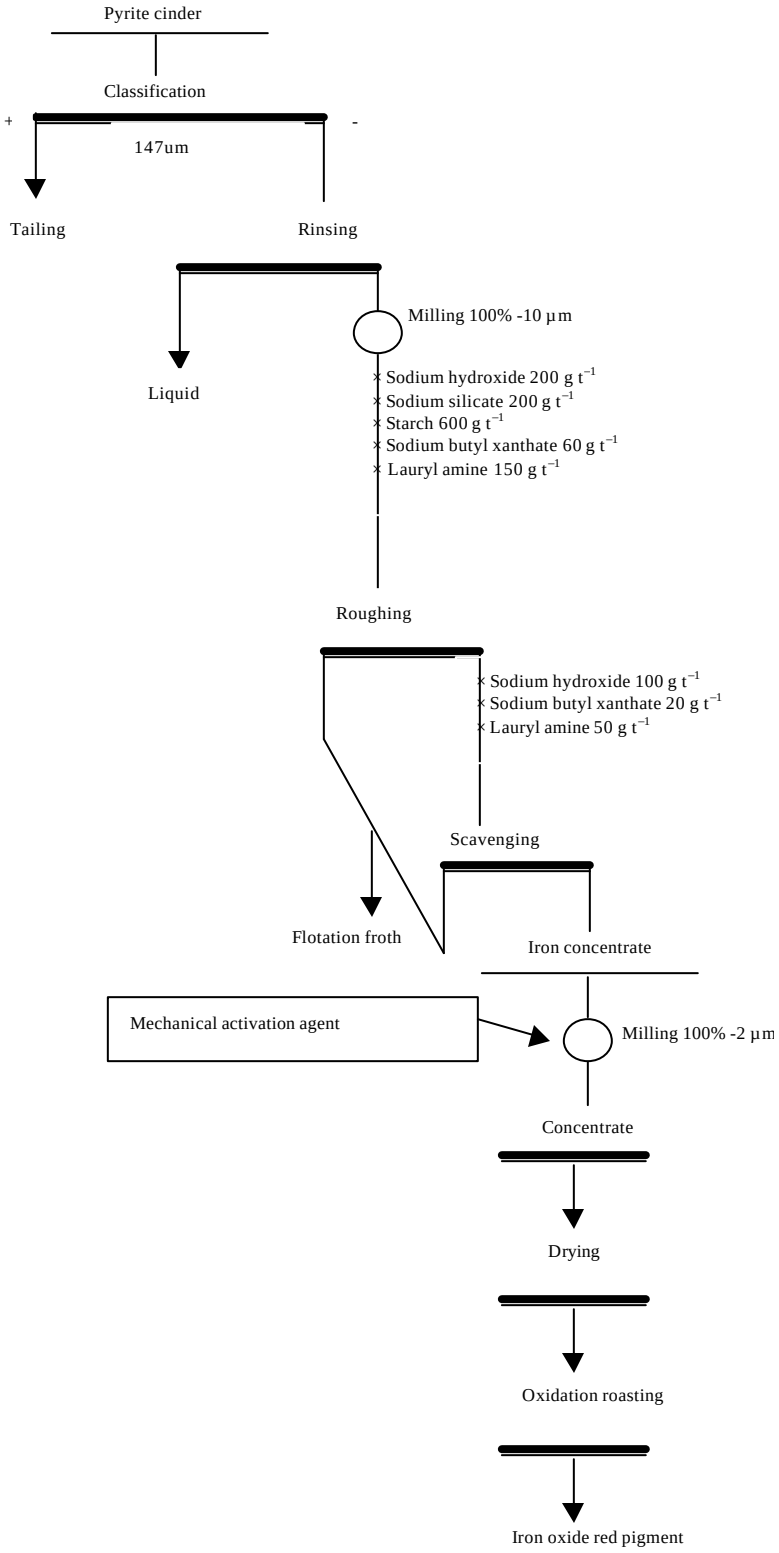

Fig. 2: Flowsheet of recovery iron oxide red pigment from pyrite cinder

Table 3: Results of entire flow circuit for recovering iron concentrate from Pyrite cinder (wt%)

		Grade			Recovery		
Production	Yield	TFe	SiO_2	S	TFe	SiO_2	S
Iron concentrate	60.18	68.65	0.68	0.06	65.96	10.03	10.73
Flotation froth	37.03	54.92	9.16	0.14	32.47	83.16	15.40
Tailing	1.51	48.89	18.18	0.77	1.18	6.73	3.45
Liquid	1.28	19.01	0.26	18.52	0.39	0.08	70.42
Feed ores	100.00	62.63	4.08	0.34	100.00	100.00	100.00

Table 4: Multi-element analysis results of iron concentrate (wt%)

Fe_2O_3	S	SiO_2	Al_2O_3	CaO	MgO	Mn	Pb	Zn	Cu	FeO
98.61	0.06	0.66	0.084	0.074	0.026	0.07	0.089	0.078	0.007	0.16

Table 5: Quality of iron oxide red prepared from pyrite cinders

Characteristic	This study	GB/T1863-2008
Colour	Red	Red
Iron content, expressed as iron (I) oxide (Fe_2O_3) determined the pigment after drying at 105°C, % (m/m) min	98.61	90
Oil absorption, % (in mass fraction)	19.3	15~25
Retative tinting strength, % (in mass fraction) min	95	90
Matter soluble in water/% (determined after drying at 105°C)(m/m)	0.3	0.5
Matter volatile at 105°C, % (m/m) max	0.2	1.5
pH of aqueous suspension	6.0	5~7
Residue on sieve of mesh aperture 63 μm, /% (m/m) max	0.1	0.5
Sum of water- soluble chlorides and sulphates expressed as the ions Cl^- and SO_4^{2-}, % (m/m) min	0.15	0.3

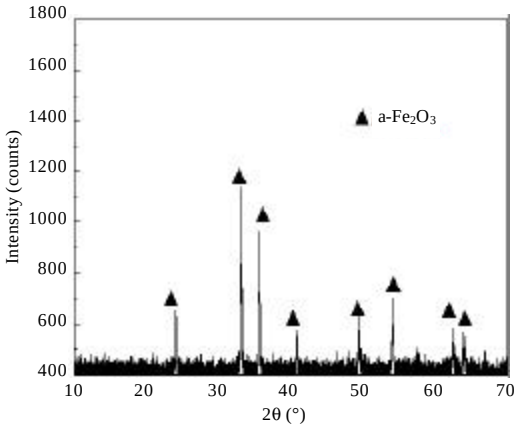

Fig. 3: XRD pattern of sample with optimal experimental parameters

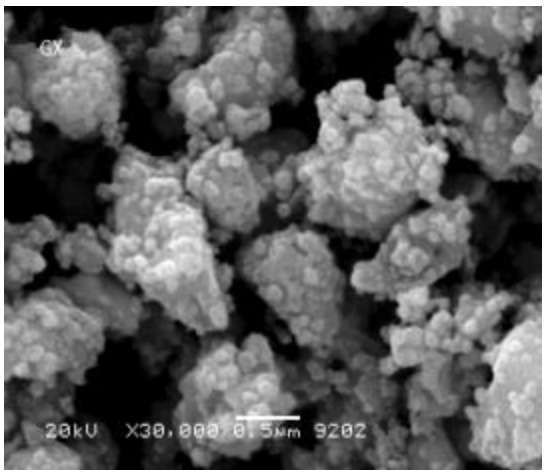

Fig. 4: SEM photographs of sample with optimal experimental parameters

Mechanical activation and oxidation roasting: An iron oxide red pigment was directly obtained by using an iron concentrate which was obtained by reverse flotation as raw materials by the process of mechanically activated by a stirring ball mill for 6 h and oxidation roasting at 600 for 1 h.

Figure 3 shows the XRD pattern of the resulting product. The results show that a single α-Fe_2O_3 phase with perfect structure was obtained with the disappearing of Fe_3O_4 phase and SiO_2 phase. The results show the physicochemical properties of an iron oxide red are changed obviously by mechanical activation and oxidation roasting under the optimal experimental conditions.

Figure 4 shows the morphology of resulting product prepared under the optimal experimental parameters. It can be seen from Fig. 4 that the shape of α-Fe_2O_3 is sphere with a mean diameter of 0.65 μm.

The result of chemical multi-element analysis of iron oxide red is shown in Table 4. It can be seen from Table 4 that the Fe_2O_3 content is 98.61% and a few contamination compounds are detected.

Waste management: In order to prevent secondary pollution, secondary waste residue (+147 μm products, flotation foam products) can be used as iron source in the

production of cement. The total waste water generated in process can meet emission standards by monitoring and adopting "lime and-basic aluminum chloride condensed" process.

CONCLUSION

Recovering iron oxide red pigment from pyrite cinders can not only control the cinders pollution, but can also supply new material for iron oxide pigment industry. Given its preferable social, economic and environmental benefits, a process consist of classification, rinsing and reverse flotation, mechanically activated and oxidation roasting was developed. The process is economical and environmentally acceptable, relatively simple and easy-to-production implementation, and the experimental results confirm its feasibility.

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