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A novel method for comprehensive utilization of sintering dust

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Abstract: A novel process aimed at the comprehensive utilization of sintering dust was developed by combining wetting grinding with sulfidization flotation. The mineralogical characteristics of the sintering dust and products were studied by powder wettability analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM) and mineral liberation analyzer (MLA). It was found that the primary lead species was laurionite and most of the particles were overwrapped with KCl. Wetting grinding was shown to accelerate the dispersion of sintering dust and transform the KCl overlay to a leachate with 20.78 g/L of K^+. A lead and silver concentrate consisting of 40.82% of Pb and 0.96 kg/t of Ag was achieved, while an iron concentrate with 60.89% of Fe was gained as tailings among sulfidization flotation. The recoveries of Pb, Ag and Fe were 89.57%, 87.85% and 88.58%, respectively. The results indicate that this method is a feasible and promising process for the comprehensive utilization of sintering dust.

Key words: sintering dust; comprehensive utilization; laurionite; wetting grinding; sulfidization flotation

1 Introduction

Steel production is associated with a significant accumulation of waste, including slag, sludge, flue dust and gases, especially in China whose crude steel output was nearly half of the world's output [1]. Among the waste, sintering dust is a solid waste generated during the sintering process in iron and steel plants which is collected by electrostatic precipitator [2,3]. It is considered to be dangerous because of the large number of heavy metals and hazardous organics it contains [4,5]. The large amount of sintering dust will occupy plenty of land and cause different degrees of environmental pollution [6−8]. Therefore, it is absolutely imperative to reutilize this waste.

Unfortunately, the comprehensive utilization rate of sintering dust is less than 20%, and most of the dust is stored at the plants [9]. The existing treatment processes can be divided into three categories: a hydrometallurgical process [10−13], a pyrometallurgical process [14,15], and a hybridization of the pyrometallurgical and hydrometallurgical processes [16,17]. Among those treatments, the most frequently used method is directly returning the sintering dust to the sintering furnace to reuse the Fe and C contained within it. However, a great amount of other elements, such as K, Na, Zn and Pb are also reused during the recycling process, some of which are harmful to normal blast furnace operations [18,19]. Water leaching is a good way to recover potassium salt from sintering dust [20,21]. However, the dispersion of sintering dust just by stirring was determined to be inefficient and the leaching residue still contains a notable amount of heavy metals.

Sulfidization flotation is a commonly used method to recover oxidized lead-based minerals, such as cerussite, anglesite, and plumbojarosite, from natural ores [22,23]. Additionally, oxidized lead minerals are frequently treated with sulfidizing agents prior to flotation [24,25]. Before flotation, grinding was used to adjust the particle size and mix solids with liquids. However, there have been few studies examining sulfidization flotation for recovering sintering dust.

In this work, a combination of wetting grinding and sulfidization flotation was applied to the separation and beneficiation of valuable elements from sintering dust. First, the composition and mineralogical characteristics of the sintering dust and products were studied by powder wettability analysis, X-ray diffraction analysis, mineral liberation analyzer and environmental scanning electron microscopy. Then, a series of studies were performed to examine the influence of wetting grinding

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on the wettability, particle size, water leaching rate, composition and morphology of the sintering dust. Finally, the sintering dust was treated with wetting grinding and sulfidization flotation under optimal conditions.

2 Experimental

2.1 Characterization of samples

The sintering dust samples were received from the sintering plant at the Baosteel Group Guangdong Shaoguan Iron & Steel Co., Ltd., China. All chemicals used in the experiments, such as sodium sulfide, starch, terpineol, 2-mercaptobenzothiazole and DDTC (sodium diethyldithiocarbamate) were of AR/GR grade. Additionally, demineralized water was used throughout the experiments.

2.1.1 Chemical analysis

A Baird PS−6 ICP-AES (inductively coupled plasma-atomic emission spectrometry, Baird Company, USA) was used to determine the trace element compositions in the solutions. Elemental analysis of the solids was performed using X-ray fluorescence (XRF) spectrometry (PANalytical Axios mAX, Netherlands).

2.1.2 Powder wettability analysis

A Mini-Lab ILMS surface science comprehensive test instrument (GBX Company, France) was used to analyze the powder wettability of the sintering dust before and after grinding. The results were obtained using the Washburn method with water as the measuring liquid. Each sample was measured five times with 1.5 g of powder.

2.1.3 Granulometric analysis

The sintering dust size distribution was determined using a Malvern Mastersizer 2000 instrument (Malvern Instruments Ltd., Worcestershire, UK) with a detection range of 0.02–2000 μm. Scattered light is detected by a detector that converts the signal to a size distribution based on the volume. The variable $D_{0.5}$ is the diameter at which 50% of the measured particle volumes were less than or equal to $D_{0.5}$. Each sample was measured five times.

2.1.4 XRD analysis

X-ray diffraction (XRD, Rigaku D/max 2500, Japan) was used to analyze the composition and crystal structure of the prepared samples at 40 kV, 100 mA for a Cu-target tube and a graphite monochromator. Standard procedures were applied to preparing the samples for analysis by X-ray diffraction [26].

2.1.5 MLA investigation

Quantitative mineralogy and microfiber studies were performed using a FEI MLA (mineral liberation analyzer) 650 system. The MLA is an automated SEM system, which identifies and quantifies the minerals based on their BSE level and compares the collected spectrum shape to a mineral data base [27−29]. This system consists of a Quanta 650 environmental scanning electron microscope, a Bruker Quantax 200 dual silicon-drift energy dispersive X-ray spectrometers using the MLA 3.1 software suite.

2.2 Wetting grinding

Wetting grinding was carried out in an XMQ *d*240 mm × 90 mm cone ball mill with a 45% steel ball filled ratio and a grinding concentration of 50%. The technical parameters consisted of a cylinder size of *d*240 mm × 90 mm, a volume of 6.25 L and a drum speed of 96 r/min. A total of 500 g sintering dust was ground in mill with 500 mL of water for several minutes at 298 K and then washed out with an additional 0.75 L of water. The solid-to-liquid ratio of the final pulp was 1:2.5. Afterwards, the pulp was filtered, dried, weighed and analyzed for iron, potassium, silver and lead content to assess the product quality and yield. The dried samples were also used for the powder wettability analysis, particles size analysis, XRD analysis and MLA investigation. Because wetting grinding is also a water leaching process, the filtered liquors were collected for multi-elemental analysis using ICP-AES (inductively coupled plasma-atomic emission spectrometry) or chemical titration.

2.3 Sulfidization flotation

The wetting grinding residues from the sintering dust were conditioned in a flotation cell for 3 min at an impeller speed of 2000 r/min. After this process, an activator was added into the flotation pulp. The pulp was then conditioned for an additional 15 min. Then, the inhibitor and collector were added into the flotation pulp and the pulp was conditioned for 5 min. Finally, a frother was added and the pulp was conditioned for an additional 2 min. The flotation tests were conducted in a 1.5 L XFG flotation cell using 375 g of leaching residue from sintering dust. The airflow rate was 2.5 L/min. All the studies were carried out with a 25% solid concentration. The concentrate and tailings were collected separately, dried, weighed and analyzed for their iron, lead and silver contents to assess the product quality and yield [30]. The wetting grinding and sulfidization flotation flowsheet for the closed-circuit sintering dust treatment is presented in Fig. 1.

3 Results and discussion

3.1 Mineralogical characterization and analysis results

3.1.1 Results of XRD analysis

The composition of the sintering dust varys widely

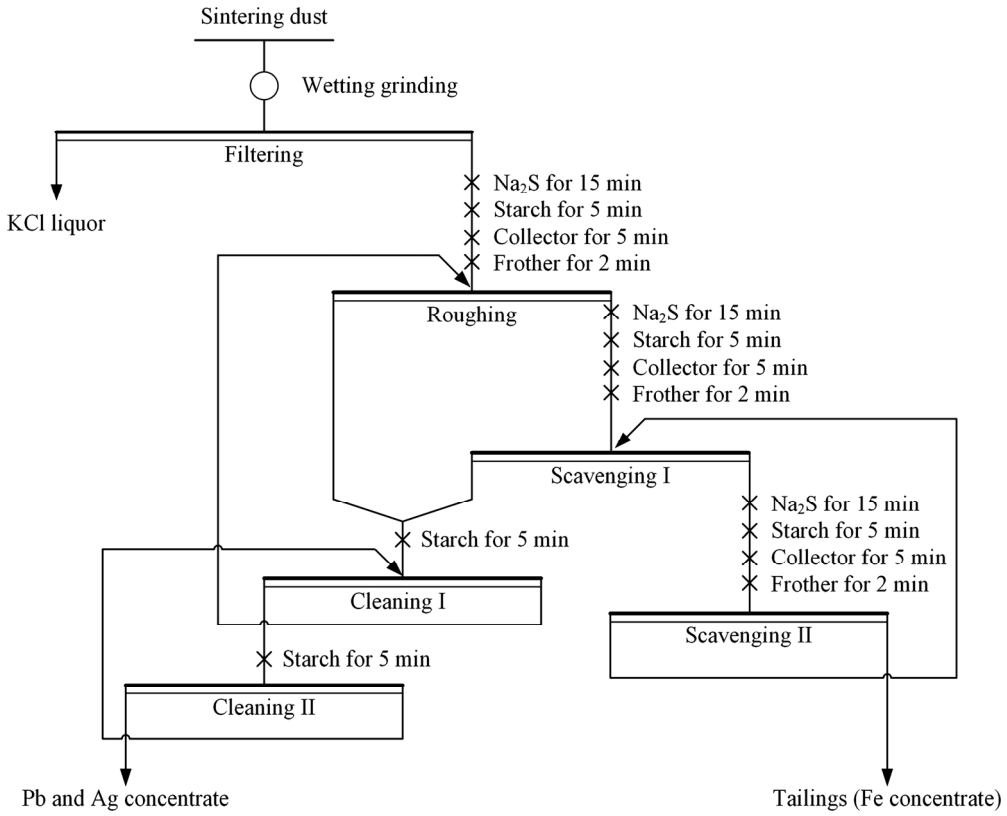

Fig. 1 Wetting grinding and flotation flowsheet for closed-circuit process of sintering dust

depending on the source, but usually contains some useful resources, such as Fe, K, Cl, Ca and Pb. Similar to previous studies [31−34], the sintering samples contain many valuable metals, such as K, Pb and Ag, which are described in Table 1. To determine the mineralogical characteristics of the sintering dust, the phase composition of the sintering dust was determined by X-ray diffraction during the initial part of the mineralogical characteristic analysis. As shown in Fig. 2(a), the main phases of the sintering dust were hematite (Fe_2O_3) and potassium chloride (KCl). The major lead bearing phase turned out to be laurionite

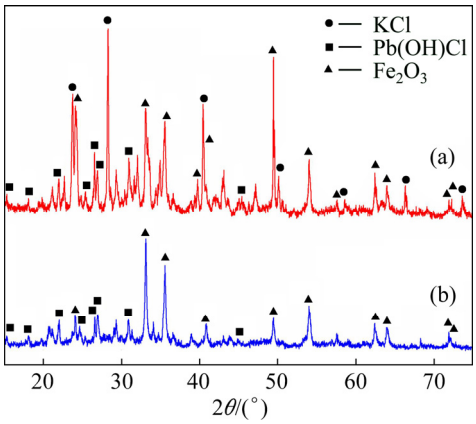

Fig. 2 X-ray diffraction patterns of sintering dust before (a) and after (b) wetting grinding

Table 1 Multi-elemental compositions of sintering dust before and after wetting grinding for 2 min (mass fraction, %) and grinding liquor (mg/L)

Product	K	Ca	Fe	Pb	Ag	Cl
Before grinding	7.24	9.13	35.60	9.16	218 (g/t)	12.15
Grinding residue	0.67	9.18	49.93	12.47	299 (g/t)	1.31
Grinding liquor	20782	3143	18	3.23	−	24590

(Pb(OH)Cl). Several phases are not marked in the figure, such as lanarikite ($Pb_2(SO)_4O$), scotlandite ($PbSO_3$), magnetite (Fe_3O_4), calcium chloride ($CaCl_2$) and gypsum ($CaSO_4·2H_2O$), due to their relatively low contents.

3.1.2 Results of MLA

Considering that a detailed mineralogical characterization investigation of the sintering dust is necessary to recover the valuable materials, mineral liberation analysis (MLA 650) was performed on the sintering dust during the second portion of the mineralogical characterization analysis. Because many basic property studies place a particular emphasis on iron and potassium [9,18−20], this work focused on the existing state of lead. Table 2 shows the MLA results of the sintering dust samples and indicates that the main lead-bearing particles are a KPbOCl-mixture, which consists of PbOHCl, KCl and has a Pb distribution rate of 54.78%. Others particles that were determined

Table 2 Mineral liberation analysis (MLA) results of lead-bearing substances

Lead bearing substance	Mass fraction/%	Formula speculated by MLA and XRD	Pb atomic distribution rate/%
Hematite	38.29	Fe_2O_3	7.43
CaFeOCl-mixture	13.20	Fe_2O_3, $CaFeO_4$, $CaCl_2$	1.77
KPbOCl-mixture	24.52	PbOHCl, KCl	54.78
Pseudocotunnite	4.60	K_2PbCl_4	24.45
KPbSOCl-mixture	2.35	$Pb_2(SO_4)O$, $PbSO_3$, KCl	7.89
Cotunnite	0.45	$PbCl_2$	3.68
Total			100

included pseudocotunnite (K_2PbCl_4), KPbSOCl-mixture (consisted of $Pb_2(SO_4)O$, $PbSO_3$ and KCl) and cotunnite ($PbCl_2$) with Pb distribution rates of 24.45%, 7.89% and 3.68%, respectively. Additionally, the hematite and CaFeOCl-mixture particles (consisting of Fe_2O_3, $CaFeO_4$ and $CaCl_2$) also had Pb distribution rates of 7.43% and 1.77%, respectively.

3.1.3 Results of SEM

To examine the surface characteristics and morphology of the sintering dust samples, research was conducted using an environmental scanning electron microscope. The SEM images of the sintering dust samples are presented in Fig. 3. Some particles had a

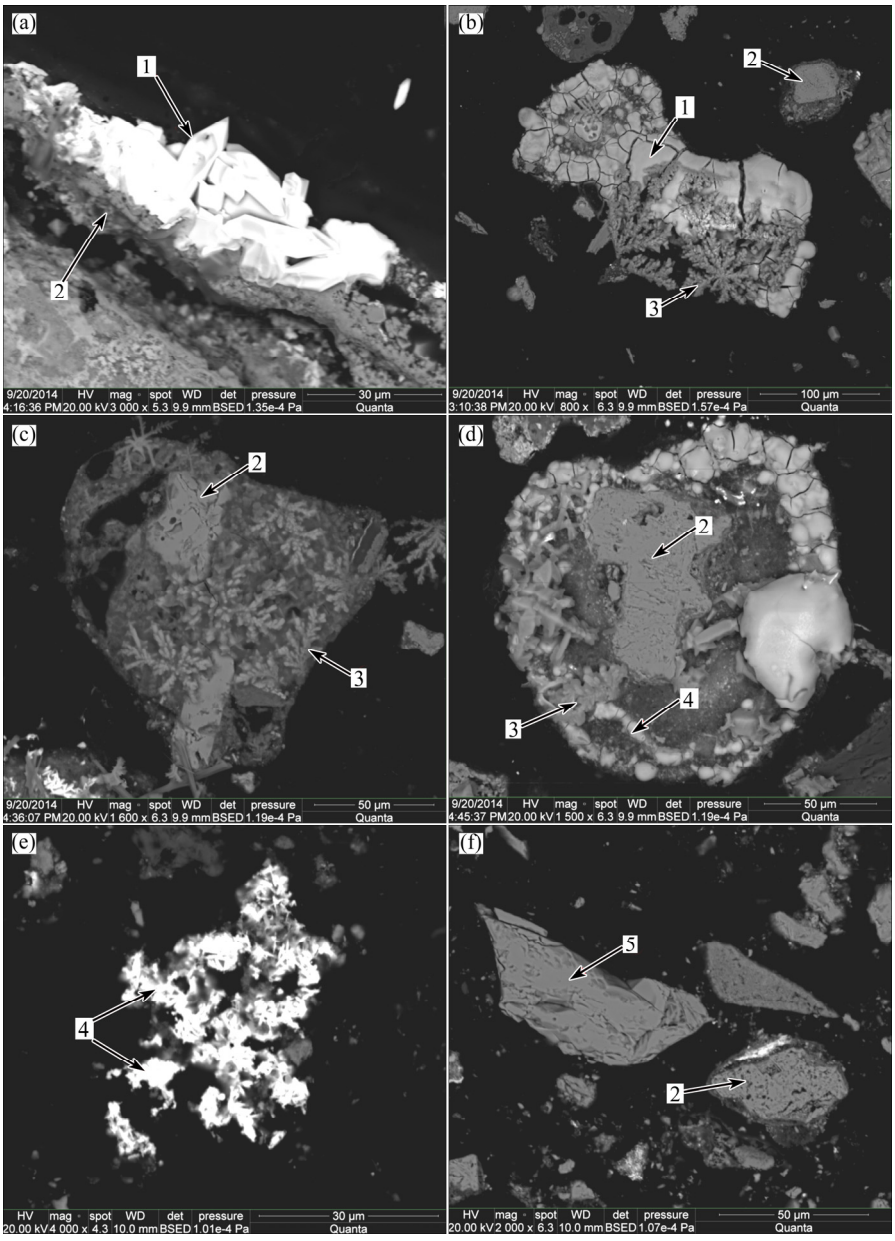

Fig. 3 SEM images of sintering dust before and after grinding: (a) Pseudocotunnite particle on hematitle particle; (b) Lead bearing particle with KCl coat; (c) Hematite particle with KCl coat; (d) Hematite particle enwrapped by lead bearing particle with KCl coat; (e) Lead-bearing particles after wetting grinding; (f) Iron-bearing particles after wetting grinding (1—Pseudocotunnite; 2—Hematite; 3—KCl; 4—Lead-bearing particle; 5—Magnetite)

clear surface morphology and integral crystal structure. As shown in Fig. 3(a), pseudocotunnite particles are associated with hematite particles.

However, most of the particles are covered by KCl, as shown in Figs. 3(b) to 3(d). The lead-bearing particles were observed to be partial overwrapped by KCl, as shown in Fig. 3(b), whereas the hematite-particles were completely overwrapped with KCl as shown in Fig. 3(c). In addition, Fig. 3(d) shows that some hematite particles are overwrapped with Pb(OH)Cl particles, which are also coated with KCl.

3.2 Wetting grinding results

The mineralogical characterization analysis results demonstrate that most of the Pb is distributed in independent minerals, such as Pb(OH)Cl and pseudocotunnite, rather than in a solid dispersion or alloy, which is the basic condition for beneficiation. Therefore, flotation is considered to be a promising method for recovering the valuable metals in the sintering dust.

Unfortunately, the KCl envelope and the inclusion of lead and iron-bearing particles creates different particles with similar surface properties, which inhibits the selective separation of the valuable particles using the flotation process. Additionally, the sintering dust is inefficiently dispersed and becomes hydrophilic, as shown in Fig. 4(a). These hydrophilic particles float without selectivity during the flotation process. Although the sample was agitated at 400 r/min for 10 min, obvious stratification of the hydrophilic and hydrophobic parts is observed, as shown in Fig. 4(c). Some researchers have shown that the dust suspension can be well dispersed in an aqueous solution through the addition of 2% sulfuric acid and 2‰ CTAB [9]. However, the addition of chemical reagents affects the following process. Therefore, a wetting grinding pretreatment was applied to the sintering dust prior to flotation.

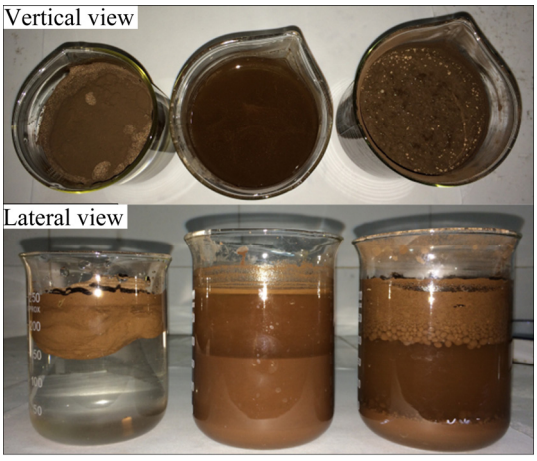

Fig. 4 Dispersibility and wettability of sintering dust under different treatments: (a) No pretreatment; (b) Wetting grinding for 2 min; (c) Agitating at 400 r/min for 10 min (every treatment has a solid-to-liquid ratio of 1:2.5 and stands for 15 min)

3.2.1 Effects of wetting grinding on wettability of sintering dust

Wetting is the first step in flotation. The effect of wetting grinding on the wettability of sintering dust was studied using the Washburn method. During the measurement, a porous tube with a filter base filled with powder comes into contact with water. The liquid is drawn up as a result of capillary action. The increase in mass of the tube, which is suspended from a force sensor, is determined with respect to time during the measurement. This process can be described by the Washburn equation:

$$\frac{m^2}{t}=\frac{C\rho^2\gamma\cos\theta}{\eta} \tag{1}$$

where m is the mass of the water absorbed, t is the time, C is a tube constant, ρ is the density of the liquid, γ is the surface tension of the liquid, θ is the contact angle of the sample and η is the viscosity of the liquid. The plot of the square of mass vs time t shows the wettability of the sintering dust. A larger ratio implies that the dust is more hydrophilic. As shown in Fig. 5, the wettability of the sintering dust increases significantly with the grinding time. In addition, the samples were well-dispersed after wetting grinding for 2 min, as shown in Fig. 4(b). Therefore, grinding can enhance the hydrophilicity and accelerate the dispersion of the sintering dust.

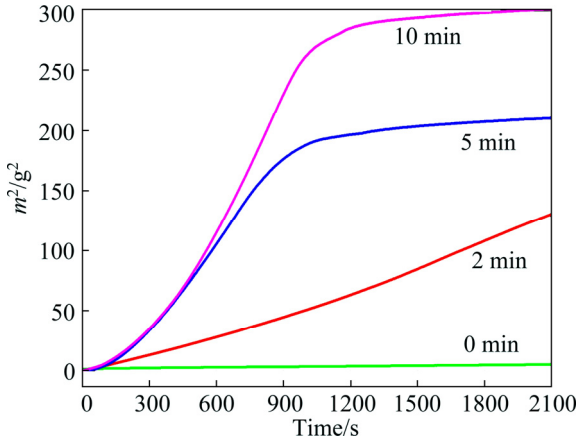

Fig. 5 Effects of wetting grinding on wettability of sintering dust

3.2.2 Effects of wetting grinding on sintering dust size

In general, the purpose of wetting grinding in beneficiation is to liberate associated minerals by reducing the particle size. However, the main aim of the wetting grinding applied in this research is to wet the sintering dust. An extremely fine particle size (less than 10 μm) causes difficulties during flotation separation.

Therefore, a granulometric distribution analysis was conducted to study the effects of grinding on sintering dust size under different grinding conditions. The results of this analysis are shown in Fig. 6. The $D_{0.5}$ of the original sintering dust (grinding for 0 min) was 20.68 μm, which indicated that physical concentration methods, such as gravity or magnetic separation, were not suitable to treat such a material. Furthermore, this size distribution suggested that the leaching kinetics of KCl in this material should be fast, due primarily to the small particle size. The $D_{0.5}$ of sintering dust samples after grinding for 2, 5 and 10 min were measured to be 15.67, 10.46 and 8.43 μm, respectively. The proportion of fine particles in the dust increased with the grinding time. Together with the mineralogical characteristics analysis, these results indicate that a proper grinding time (such as 2 min) is beneficial to the liberation of particles, whereas a long grinding time has a negative effect on the flotation results due to the increased number of fine particles.

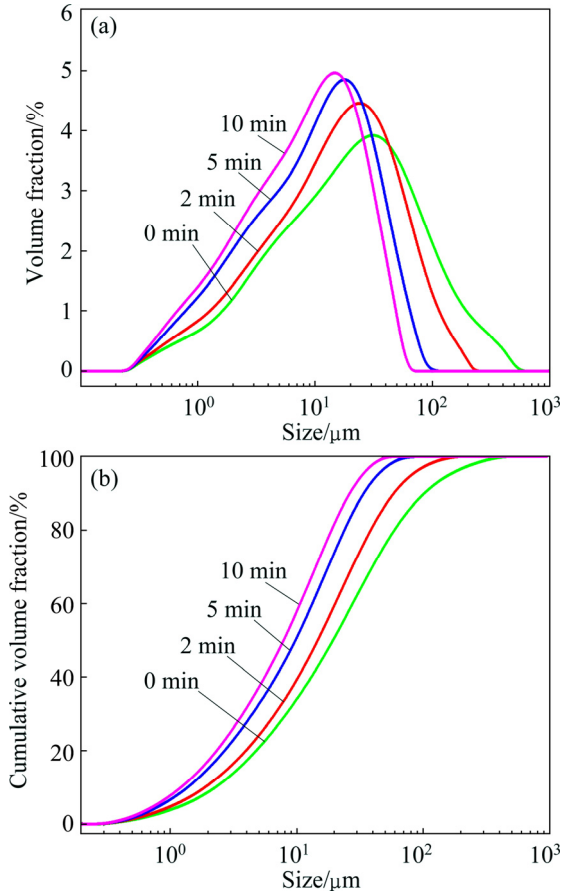

Fig. 6 Effects of wetting grinding on size of sintering dust

3.2.3 Effects of wetting grinding on water leaching of sintering dust

Figure 7 shows the effect of grinding on the grade of the Cl and K in the residue and indicates that solubility equilibrium for the chlorine and potassium could be achieved within 5 min. The content of K in the residue reduced significantly from 7.24% to 0.67% for a leaching rate of 92.06% over 2 min. The content of Cl also experienced a similar sharp decline from 12.15% to 1.31% for a leaching rate of 90.78% over 2 min. When the grinding time increased from 2 to 10 min, the content of K and Cl exhibited a slight decrease to 0.58% and 1.24% over 5 min, respectively, and 0.50% and 1.21% over 10 min, respectively. The high dissolution speed was due to the small particle size and high solubility of the potassium chloride. Additionally, the KCl liquor had a potassium ion concentration of 20.78 g/L, a calcium ion concentration of 3.14 g/L and a chloridion concentration of 24.59 g/L. This solution was suitable to extract potassium, as a series of studies have been indicated [9,19−21,35].

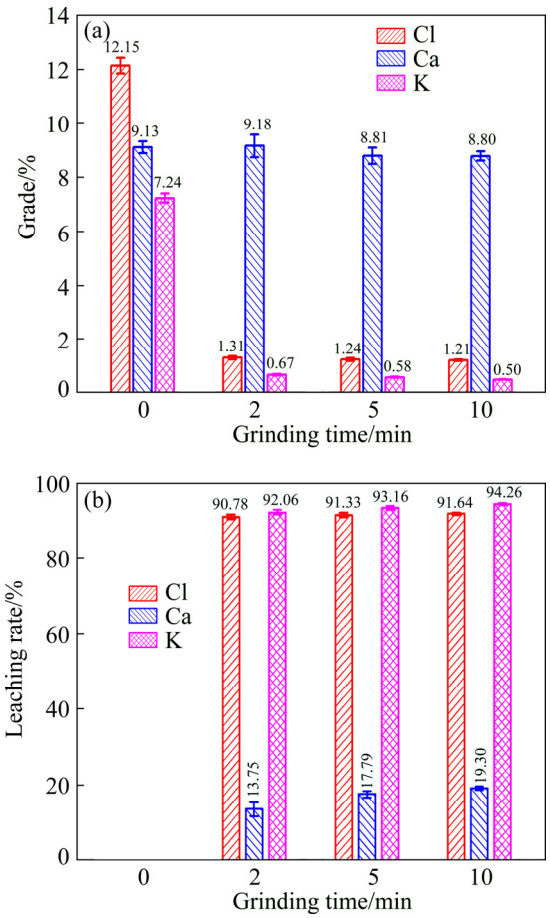

Fig. 7 Effects of wetting grinding on residual grade (a) and leaching rate (b) of Cl, Ca and K in sintering dust

With the exception of KCl, there are other dissolved constituents because 26.94% of the sintering dust was dissoluble after grinding for 2 min. This result is confirmed from the elemental composition of colatuie after grinding, as shown in Table 1. This result also demonstrated that there are other chlorides in the sintering dust, such as calcium chloride.

However, the dissolution speed of Ca is slower than those of Cl and K. As shown in Fig. 7, the content of Ca in the residue increased from 9.13% to 9.18% after 2 min and then reduced to 8.81% after 5 min and 8.80% after 10 min. The dissolution rates were 13.75%, 17.79% and 19.30%, respectively, and increased with time. The differences in the dissolution speed and rate were due to the KCl covering on the surface of most of the particles (including the calcium bearing particles) and the low solubility of the calcium containing compounds. Additionally, 2 min of grinding causes the filter liquor to have few calcium ion impurities.

Other elements contained in the residue have also been altered. As shown in Table 1, the contents of Fe and Pb in the sintering dust sample increase from 35.60% and 9.16% to 49.93% and 12.47%, respectively, whereas the Ag content increases from 218 g/t to 299 g/t. These valuable metals are enriched after wetting grinding due to the dissolution of KCl and other soluble substances. Additionally, an overall consideration of the effects of grinding on wettability, particle size and water leaching indicated that a grinding time of 2 min was optimal for the water leaching of KCl and the wetting of the sintering dust.

3.2.4 Effects of wetting grinding on composition and morphology of sintering dust

Because wetting grinding changes the elemental compositions in sintering dust, it may also influence the phases and morphology of the sintering dust. To identify this influence, XRD and SEM analyses were conducted on the wetting grinding residue. The XRD analysis of the residue (shown in Fig. 2(b)) revealed that the composition of the samples was simplified after grinding. The major components are primarily Fe_2O_3 and Pb(OH)Cl, whereas KCl identified prior to wetting grinding was not found. The SEM images of the material after grinding are shown in Figs. 3(e) and (f). The surfaces of lead- and iron-bearing particles are exposed and the KCl cladding is absent.

3.3 Sulfidization flotation results

The regulation of agents used in the sulfidization flotation is as follows. The sulfidizing agent was sodium sulfide, which also acted as the activator for the lead-bearing particles. The dosage of the sodium sulfide was 2 kg/t. Starch was used as the inhibitor for the iron-bearing particles with a dosage of 4 kg/t. The collector was a mixture of sodium diethyldithiocarbamate and 2-mercaptobenzothiazole. The dosages of these compounds were 800 g/t and 200 g/t, respectively. Additionally, the frother was terpineol with a dosage of 150 g/t.

3.3.1 Flotation separation results

The feed from the sulfidizaton flotation was the residue of sintering dust after wetting grinding for 2 min. A slurry pH of 8−9 was maintained throughout the experiment during the hydrolysis of sodium sulfide. As shown in Table 3, the flotation concentrate consisted of lead and silver. The grade and recovery of Pb were 40.82% and 89.57%, respectively, and the grade and recovery of Ag were 960 g/t and 87.85%, respectively. The tailings, which consisted of the iron concentrate, contained 60.89% Fe and 1.79% Pb. The recovery of Fe was 88.58%, which was suitable for directly returning to the iron fabrication process.

3.3.2 Effect of flotation on elemental distribution in products

Table 4 shows the effect of flotation on the elemental distribution in the concentrates and tailings. These results indicated that flotation also affected the separation of fluorine, silicon, chlorine and calcium-bearing particles in the leaching residue. The distribution in the Pb and Ag concentrates was 24.18% for Si, 39.02% for Ca, 45.84% for S, 46.03% for F and 80.12% for Cl, which reduced impurities in the Fe concentrate. Additionally, 71.91% of Cu in the sintering dust was enriched in the flotation concentrate, which can be comprehensively recovered using the pyro-metallurgical or hydrometallurgical process employed for the lead concentrate.

Table 3 Results of closed-circuit flotation with wetting grinding residue of sintering dust

Product	Yield/%	w(Fe)/%	w(Pb)/%	w(Ag)/(g·t^{-1})	Recovery/%		
					Fe	Pb	Ag
Pb and Ag concentrate	27.36	20.84	40.82	960	11.42	89.57	87.85
Tailing (Fe concentrate)	72.64	60.89	1.79	50	88.58	10.43	12.15
Feed (Grinding residue)	100	49.93	12.47	299	100	100	100

Table 4 Effects of flotation on elements distribution in concentrate and tailing

Product	Distribution rate/%								
	Fe	Si	Ca	S	F	Cu	Cl	Ag	Pb
Pb and Ag concentrate	11.42	24.18	39.02	45.84	46.03	71.91	80.12	87.85	89.57
Tailing (Fe concentrate)	88.58	75.82	60.98	54.16	53.97	28.09	19.88	12.15	10.43

4 Conclusions

1) The mineralogical characterization analysis of the sintering dust revealed that the primary species of lead in the dust was laurionite, and most of the particles were overwrapped with potassium chloride.

2) Wetting grinding played a significant role in the utilization of sintering dust by increasing the wettability and accelerating the dispersion of the sintering dust. After wetting grinding, the contents of Fe and Pb in sintering dust samples upgraded from 35.60% and 9.16% to 49.93% and 12.47%, respectively, whereas that of Ag increased from 218 g/t to 299 g/t. Meanwhile, the KCl overwrap was removed and a leaching liquor containing 20.78 g/L K^+ with K leaching rate of 92.06% was achieved.

3) The following sulfidization flotation was effective. The flotation sulfidization concentrate consisted of 40.82% Pb and 0.96 kg/t Ag with recoveries of 89.57% and 87.85%, respectively. Additionally, the tailings consisted of the Fe concentrate containing 60.89% of Fe and an iron recovery of 88.58%.

4) By combining wetting grinding with sulfidization flotation, a promising processing method for the comprehensive utilization of sintering dust can be achieved, which not only reduces the amount of waste and saves disposal cost, but also provides raw materials for the metallurgy of K, Fe, Pb and Ag.

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一种烧结机静电除尘灰综合回收的新方法

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摘 要：提出一种采用润湿球磨和硫化浮选相结合的综合回收烧结机静电除尘灰的新方法。采用粉末润湿性分析、X 射线衍射(XRD)、扫描电子显微镜(SEM)和矿物参数自动定量分析系统(MLA)研究烧结机静电除尘灰及处理产物的矿物学特性。结果表明：烧结机静电除尘灰主要的含铅物相是羟基氯化铅，同时大部分的颗粒都被氯化钾包覆。润湿球磨能够加速烧结机静电除尘灰的分散，同时氯化钾包被转化为 K^+浓度为 20.78 g/L 的浸出液。硫化浮选过程中可以得到 Pb 品位 40.82%、Ag 品位 0.96 kg/t 的铅银精矿以及 Fe 品位 60.89%的铁精矿。Pb、Ag 和 Fe 的回收率分别达 89.57%、87.85%和 88.58%。研究结果显示，提出的新方法是一种可行且有前景的烧结机静电除尘灰综合利用方法。

关键词：烧结机静电除尘灰；综合利用；羟基氯化铅；润湿球磨；硫化浮选

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