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Study on Enhancing Zinc Dusts with Direct Sulphuric Acid Leaching

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The mechanical activation of zinc dusts to enhance leaching of zinc and lead going into leaching residue with direct sulphuric acid leaching have been performed in the paper. Some technical parameters were mainly investigated and optimized, including concentration of initial acid, liquid-solid ratio, leaching time and leaching temperature. The SEM measurement and DTA-TG analysis were conducted to check the change of microstructure and thermodynamics stability of zinc dusts before and after mechanical activation for 0.5 h. It was found that before mechanical activation the optimum technical conditions were concentration of H_2SO_4 175 g/L, liquid-solid ratio 7:1, leaching temperature 80 °C, leaching time 60 min and leaching rate of zinc was 92.47 % and rate of lead going into leaching residue was 90.83 %. After 0.5 h milling, the optimum technical parameters were concentration of H_2SO_4 150 g/L, liquid-solid ratio 5:1, leaching temperature 50 °C, the leaching time 40 min and leaching rate of zinc was 91.52 % and rate of lead going into leaching residue was 95.36 %.

Keywords: Zinc dusts, Leaching rate, Mechanical activation, Lattice distortion.

INTRODUCTION

Recently the galenite and blende resources become increasingly scarce which used as the main raw material of lead and zinc smelting with the rapid development of zinc and lead smelting industries. Therefore, exploitation and utilization of secondary resources and the comprehensive recovery and utilization of waste bearing zinc and lead has attracted more and more attention[1-4]. Large amounts of zinc dusts were produced during fuming stage of lead smelting with the Ausmelt technology. The content of zinc and lead is up to about 75 wt. %. Meanwhile, there are a small amount of hazardous elements such as arsenic, antimony and sulphur. Simple piling up of these dusts will bring environmental pollution. Therefore reasonable recycling of zinc and lead dusts will generate good economic and environmental benefits[5,6].

In recent years, with the development of mechanical chemistry it has been gradually recognized that mechanical activation can induce changes of crystal structure and physicochemical properties of minerals such as lattice defects and non-crystallization which can increase internal energy and reaction activity of minerals[7-10]. The mechanical activation has been applied widely in enhancement leaching of difficult leaching minerals to recover valuable metals effectively[11-14].

In this paper, direct sulphuric acid leaching zinc dusts before and after mechanical activation were contrastively investigated for the first time. The effects of some technical parameters on the leaching rate of zinc and going into leaching residue rate of lead were studied. At last the parameters of zinc and lead dusts leaching process were optimized.

EXPERIMENTAL

The experiment materials were provided by some lead smelter of Yunnan province and the main chemical composition was listed in Table-1. The leaching reagents are formulated with different concentrations of H_2SO_4 solution. X-ray diffraction analysis shows that the main phases of zinc are zinc oxide and metallic zinc and those of lead are lead oxide and lead arsenate.

The dusts before mechanical activation were dried at 80 °C for 5 h and then sieved by 60 mesh and -60 mesh particles were used as experimental materials. The mechanical activation was conducted in a planetary-type ball mill(XQM-0.4L with four PTFE vessels and some agate balls with different sizes, Nanjing University Instrument Plant, Nanjing, China). The process parameters of mechanical activation were fixed as follows: the ratio of ball to material mass was 7:1, the number of big ball (24 mm in diameter) to small ball (10 mm in diameter) was 1:6, the rotation speed was 300 rpm and the activation time was 0.5 h.

The leaching experiments were carried out in glass containers. The thermostatic magnetic stirrer was used to either

TABLE-1
CHEMICAL COMPOSITION OF ZINC AND LEAD DUSTS

Chemical composition	Pb	Zn	As	S	Cd	Cl	Sb	SiO_2	Fe	Al_2O_3
Content (wt. %)	39.07	33.71	2.99	2.38	0.512	0.293	0.288	0.267	0.25	0.215

control reaction temperature or stir reaction mixture. The vacuum filtration equipment was used to separate leaching solution and leaching residue.

The leaching rate of zinc and the leaching residue rate of lead were analyzed by EDTA chelatometry. The morphology and microstructures analysis of dusts before and after mechanical activation were performed using a scanning electron microscope(Philips XL-30ESEM). The differential thermal analysis and thermo-gravimetry analysis was detected by high temperature thermal analyzer (ZRY-2P) with the parameters as follows: the heating rate is 10 °C/min, the testing temperature range is from 50 to 1200 °C and the nitrogen gas flow rate is 60 mL/min.

RESULTS AND DISCUSSION

Fig. 1 shows SEM micrographs of experimental raw materials before and after mechanical activation for 0.5 h. It can be seen that the raw materials aggregates into polygonal bulks before being milled shown in Fig. 1a. After mechanical activation for 0.5 h, the microstructure has a significant change, the irregular bulks becomes fragmentation, refinement, well-distribution, surface roughening and covering some loose floccules as shown in Fig. 1b, which is consistent with literatures[10,11,13].

The effect of the concentration of H_2SO_4 on leaching rate of zinc and the rate of lead going into leaching residue was shown in Table-2 and Fig. 2. As shown in Fig. 2, the effect of H_2SO_4 concentration on leaching rate of zinc and lead going into leaching residue are obtained before mechanical activation and after mechanical activation for 0.5 h. Fig. 2a b show the leaching rate of zinc and lead going into leaching residue increased with the increasing of the concentration of H_2SO_4, while the trend of augment declined with further increasing of the concentration. With the concentration of H_2SO_4 increasing to 175 g/L, the leaching rate of zinc was up to 90.21 % and of lead going into residue was up to 92.01 %. While the leaching rate of zinc reached 94.06 % and the rate of lead going into residue was up to 96.58 % when the concentration of H_2SO_4 is 150 g/L after mechanical activation for 0.5 h. It demonstrated that the conclusion that the mechanical activation can reduce the dependence of leaching rate of zinc and the rate of lead going into leaching residue on the concentration of H_2SO_4.

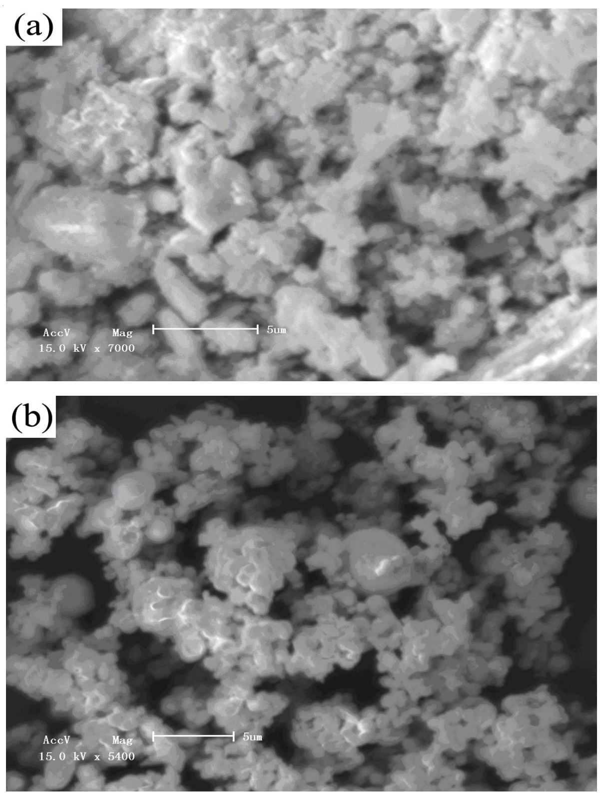

Fig. 1. SEM images of experimental materials before mechanical activation (a) after mechanical activation for 0.5 h (b)

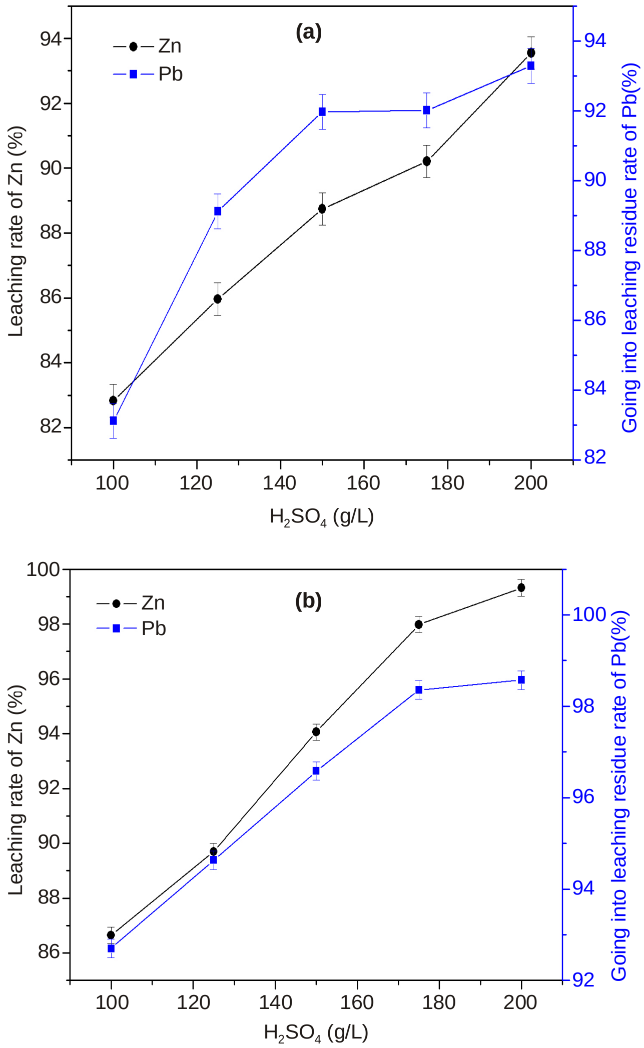

Fig. 2. Effect of H_2SO_4 concentration on leaching rate of zinc and the rate of lead going into leaching residue: (a) before mechanical activation, (b) after mechanical activation for 0.5 h

TABLE-2
EFFECT OF H_2SO_4 CONCENTRATION ON LEACHING RATE OF ZINC AND THE RATE OF LEAD GOING INTO LEACHING RESIDUE

Concentrations of H_2SO_4 (g L^{-1})	100	125	150	175	200
Leaching rate of zinc (before mechanical activation) (%)	82.83	85.96	88.74	90.21	93.55
Leaching rate of zinc (after mechanical activation 0.5 min) (%)	86.65	89.70	94.06	97.98	99.32
Leaching rate of lead going into leaching residue (before mechanical activation) (%)	83.12	89.12	91.97	92.01	93.29
Leaching rate of lead going into leaching residue (after mechanical activation 0.5 min) (%)	92.70	94.63	96.58	98.36	98.57

The effect of the liquid-solid ratio on leaching rate of zinc and the rate of lead going into leaching residue is presented in Table-3 and Fig. 3. It can be seen from Fig. 3 that with the increasing of the liquid-solid ratio the leaching rate of zinc and lead going into leaching residue increased. The leaching rate of zinc was up to 90.91 % and the rate of lead going into residue was up to 91.50 % when the liquid-solid ratio was 7:1 before mechanical activation. However, after milling the dusts for 0.5 min the leaching rate of zinc was up to 96.38 % and of lead going into residue was up to 95.86 % when the liquid-solid ratio was 5:1. The mechanical activation can reduce the dependence of leaching rate of zinc and the rate of lead going into leaching residue on the liquid-solid ratio dramatically.

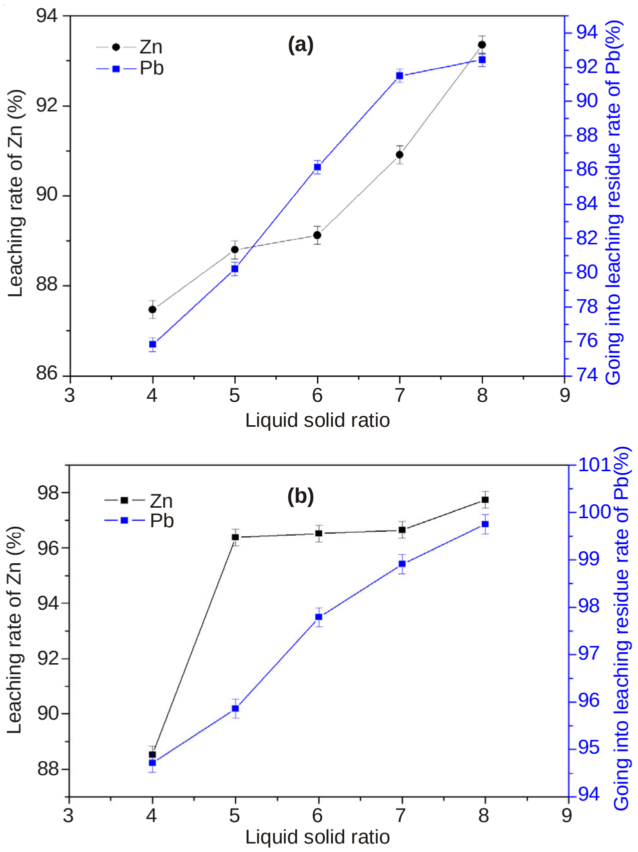

Fig. 3. Effect of liquid-solid ratio on leaching rate of zinc and the rate of lead going into leaching residue: (a) before mechanical activation, (b) after mechanical activation for 0.5 h

Table-4 and Fig. 4 illustrate the effect of the leaching temperature on leaching rate of zinc and the rate of lead going into leaching residue. As shown in Fig. 4, the leaching rate of zinc and of lead going into leaching residue increased with the leaching temperature but the increase extent were not obvious. When the temperature increasing to 80 °C, the leaching rate of zinc was up to 92.03 % and of lead going into residue was up to 91.52 % before mechanical activation, respectively. However, when the dust was milled for 0.5 h the leaching rate of zinc was up to 94.32 % and of lead going into residue was up to 93.50 % when the leaching temperature was set as 50 °C. The mechanical activation can reduce the

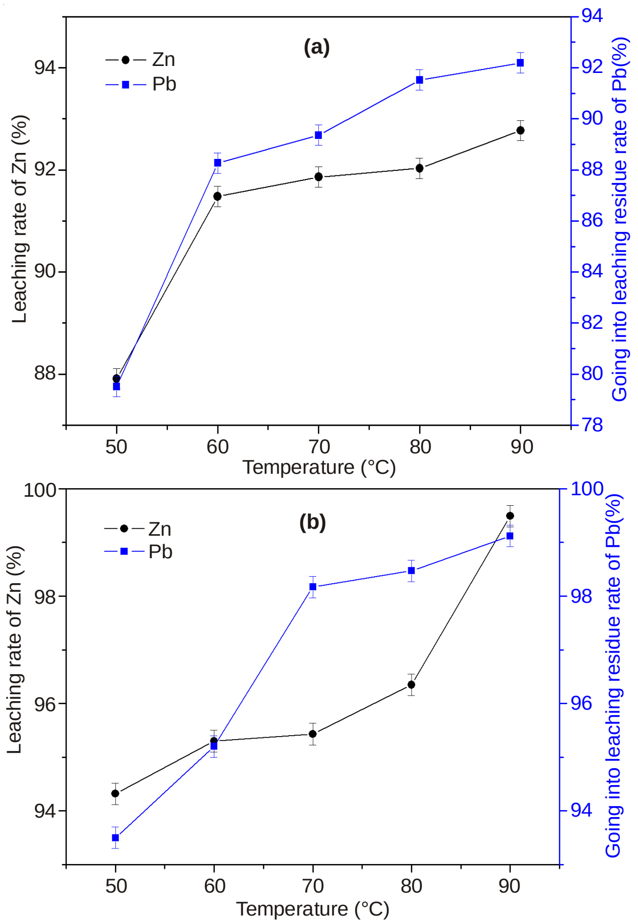

Fig. 4. Effect of leaching temperature on leaching rate of zinc and the rate of lead going into leaching residue: (a) before mechanical activation, (b) after mechanical activation for 0.5 h

TABLE-3
EFFECT OF LIQUID-SOLID RATIO ON LEACHING RATE OF ZINC AND THE RATE OF LEAD GOING INTO LEACHING RESIDUE

Liquid-solid ratio	4:1	5:1	6:1	7:1	8:1
Leaching rate of zinc (before mechanical activation) (%)	87.47	88.80	89.12	90.91	93.35
Leaching rate of zinc (after mechanical activation 0.5 h) (%)	88.53	96.38	96.52	96.65	97.74
Leaching rate of lead going into leaching residue (before mechanical activation) (%)	75.82	80.23	86.17	91.50	92.43
Leaching rate of lead going into leaching residue (after mechanical activation 0.5 h) (%)	94.72	95.86	97.79	98.91	99.75

TABLE-4
EFFECT OF LEACHING TEMPERATURE ON LEACHING RATE OF ZINC AND THE RATE OF LEAD GOING INTO LEACHING RESIDUE

Leaching temperature (°C)	50	60	70	80	90
Leaching rate of zinc (before mechanical activation) (%)	87.91	91.48	91.86	92.03	92.77
Leaching rate of zinc (after mechanical activation 0.5 min) (%)	94.32	95.30	95.43	96.35	99.49
Leaching rate of lead going into leaching residue (before mechanical activation) (%)	79.52	88.27	89.36	91.52	92.19
Leaching rate of lead going into leaching residue (after mechanical activation 0.5 min) (%)	93.50	95.20	98.17	98.47	99.12

dependence of leaching rate of zinc and of lead going into leaching residue on the leaching temperature.

The effect of leaching time on leaching rate of zinc and of lead going into leaching residue are given in Table-5 and Fig. 5. Before milling the leaching rate of zinc and of lead going into leaching residue increases with the leaching time but the increase extent is not obvious. When the time increasing to 60 min, the leaching rate of Zn was up to 92.47 % and of lead going into residue was up to 90.83 % before mechanical activation, respectively. However, when the dusts were mechanical activated for 0.5 h the leaching rate of Zn was up to 91.52 % and of Pb going into residue was up to 95.36 % when the leaching time was set as 40 min. The mechanical activation can reduce the dependence of leaching rate of zinc and the rate of lead going into leaching residue on the leaching time dramatically.

Fig. 6, represents the differential thermal analysis-thermogravimetric (DTA-TG) curve of lead and zinc dusts before and after milling for 0.5 min. As shown, before mechanical activation the TG curve keeps constant and there is no mass loss before 850 °C. There was a obvious mass loss at about 900 °C, which is due to the decomposition or volatilization of some chemical components in the dust. After being milled for 0.5 min, the TG curve has a similar change law. While the initial temperature shifts left and the mass loss rate increases slightly. It may be ascribed that mechanical activation can reduces thermal stability and increases the reaction activity of the experimental materials. Corresponding with the mass loss, the DTA curve of the materials before being milled there is an endothermal peak at about 100 and 800 °C. The first endothermic peak represents phase change or crystal form change reaction. The second endothermal peak according with a much mass loss demonstrates that there is a volatilization or decomposition reaction. After mechanical activation for 0.5 h the endothermic peak position shifts to the lower temperature and the peak value increases which show that in process of mechanical ball milling with the destruction of crystal structure which is consistent with the literatures[9,11].

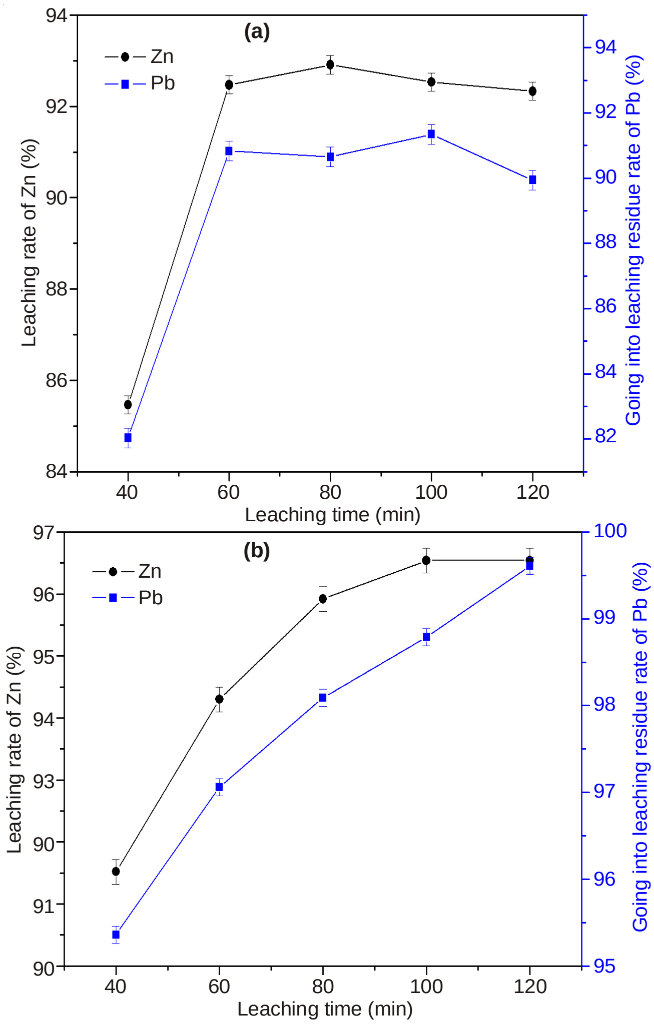

Fig. 5. Effect of leaching time on leaching rate of zinc and the rate of lead going into leaching residue: (a) before mechanical activation, (b) after mechanical activation for 0.5 h

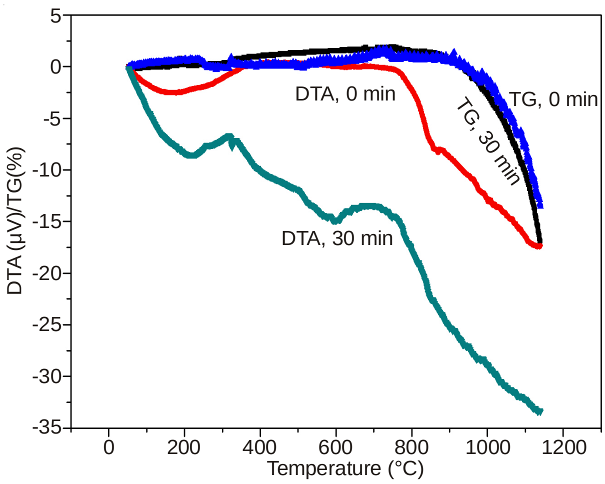

Fig. 6. DTA-TG curve of lead and zinc dusts before and after milling for 0.5 h

TABLE-5
EFFECT OF LEACHING TIME ON LEACHING RATE OF ZINC AND THE RATE OF LEAD GOING INTO LEACHING RESIDUE

Leaching time (min)	40	60	80	100	120
Leaching rate of zinc (before mechanical activation) (%)	85.46	92.47	92.91	92.53	92.33
Leaching rate of zinc (after mechanical activation 0.5 min) (%)	91.52	94.30	95.92	96.54	96.54
Leaching rate of lead going into leaching residue (before mechanical activation) (%)	82.03	90.83	90.65	91.34	89.94
Leaching rate of lead going into leaching residue (after mechanical activation 0.5 h) (%)	95.36	97.06	98.09	98.79	99.61

Conclusion

The optimum technical parameter of the zinc dusts leaching by sulphuric acid before mechanical activation was that the H_2SO_4 concentration 175 g/L, the liquid-solid ratio 7:1, the leaching temperature 80 °C and the leaching time was 60 min, at the optimum condition the leaching rate of zinc is 92.47 % and of lead going into leaching residue is 90.83 %. The optimum technical parameter of the zinc dusts leaching by sulphuric acid after mechanical activation for 0.5 h was that the H_2SO_4 concentration 150 g/L, the liquid-solid ratio 5:1, the leaching temperature 50 °C and the leaching time was 40 min, at the optimum condition the leaching rate of zinc is 91.52 % and the rate of lead going into leaching residue is 95.36 %, respectively. The mechanical activation can make experimental materials fragmentation, refinement, well-distribution, surface roughening, destruct the crystal structure, reduce thermal stability and increase the reaction activity. Therefore, the mechanical activation can reduce the dependence of leaching rate of zinc and of lead going into leaching residue on the initial acid concentration, the liquid-solid ratio, the leaching temperature and the leaching time dramatically.

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