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Technical Note

Leaching of Willemite Concentrate in Sulfuric Acid Solution at High Temperature

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Abstract: The technical practicability of high temperature leaching process using willemite concentrate as the materials was developed in the study for extracting base metals. The factors affecting the leaching rates of zinc, iron, and silica from willemite concentrate were investigated, and optimized for maximum zinc extraction and minimum iron and silica dissolution to meet the needs of solution purification. Under the studied conditions (413 K, 0.81 mol/L sulfuric acid, duration 60 min, liquid to solid ratio (L/S) 10 mL/g, agitation speed 550 rpm, particle size of −250 + 300 mesh and 1.4 MPa pressure), up to 97% zinc was leached into lixivium together with approximate 5.72% SiO_2 and 21.11% Fe. The performance of liquid-solid separation was also improved with decreasing silica dissolution under the experimental conditions used.

Keywords: high temperature leaching; willemite; zinc extraction; removing impurities

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1. Introduction

Metallic zinc as an important member of the nonferrous metals is produced from the secondary zinc resource, its sulfide ores, oxide ores and silicate ores using pyro- and/or hydrometallurgical processes [1–4]. The modern extractive metallurgy of zinc is dominated by the roasting-leaching-Zn electrowinning process [2,3]. As the main zinc resources (like high grade ZnS ores) deplete rapidly, and the metallic zinc demand increases quickly, the siliceous types of zinc resources as an alternate resource are becoming more attractive [4,5]. They generally contain zinc in the form of hemimorphite and willemite as well as high content of silica, and are a very rarely used without available cost-efficient production technologies [6,7].

For siliceous zinc ores, the pyrometallurgical process is likely to be losing in importance progressively due to high consumption of energy [7]. The use of solar technology as a new pyrometallurgical attempt was studied for the extraction of zinc from willemite by Weidenkaff et al. [8] who found that the dissociation reaction of natural zinc ores can occur by using solar energy in the solar furnace. In the hydrometallurgical process, there are main three kinds of different leaching systems available for extraction of zinc from siliceous zinc ores, i.e., acid leaching system [4,7,9–15], alkaline media [16–19] and bioleaching processes [5]. In the said method, sulfuric acid leaching is a well known process and its application has been employed in some commercial plants to the recovery of zinc from siliceous zinc ores, but alkaline- and bio-leaching processes have also a promising future.

Direct acid leaching reactions for hemimorphite and willemite present in the siliceous zinc ores can be given according to Equations (1) and (2):

$$Zn_4Si_2O_7(OH)_2 \cdot H_2O + 4H_2SO_4 \rightarrow 4ZnSO_4 + Si_2O(OH)_6 + 3H_2O \quad (1)$$

$$Zn_2SiO_4 + 2H_2SO_4 \rightarrow 2ZnSO_4 + Si(OH)_4. \quad (2)$$

It is important to note that the leached silica, if not handled appropriately, will change to silicon gel that brings about the gelatinization of lixivium or obviously increases the viscosity of leaching solution. The above phenomenon damages or totally interrupts the pulp filtration and restricts the relative movement between liquid phase and solid phase in the slurry, resulting in decrease of the extraction of zinc into leaching solution.

For overcoming the said problems, a number of methods had been directly studied to treat zinc silicate ores. Dufresne [9] developed a quick leaching process according to the chemistry of a water-starved system to deal directly with zinc silicate ores. Abdel Aal and Shukry [20] studied the leaching of Umm Gheig area (Egypt) zinc silicate ores with said technique. Matthew and Elsner [10] reported that in a consecutive leaching system, silica was firstly dissolved into leaching solution, and then curdled by adjusting the pH of leaching solution. The lixivium pH was adjusted to pH 4–5.5 using neutralization reagent for precipitation and coagulation of silica sol. Sulfuric acid leaching of zinc silicate ores (Thailand) was studied by using Magnafloc 156 as the flocculating agent [12]. It was reported that the leaching rates of zinc reach 95% under the condition of sulfuric acid concentration 4.5 M, liquid-solid ratio (L/S) 10:1 mL/g, leaching time 3 h, and temperature 343 K, and that a small amount of flocculating agents (0.5 g/kg of ore) were used in comparison with the other one. Hua and Zhang et al. [7] studied the leaching of a Yunnan Province (China) zinc silicate ore with microwave radiation. It was found that the impurities (such as silica and iron) present in the leaching solution could be effectively removed by using microwave radiation, and that zinc extraction rate was up to 99.08%, and silica and iron dissolution rates reached a low of 0.30% and 0.10% under the studied conditions. The Skorpion sulphuric acid leach/solvent extraction process was reported by Gnoinski [13] to leach zinc from non-sulfide ores. The silica arriving from leaching solution was successfully precipitated by using $CaCO_3$. Iron can't be easily removed from the organic phase during solvent extraction. It was stated by Souza et al. [14] that an integrated process was industrially applied to dispose of zinc silicate concentrates and zinc sulfide concentrates at the Três Marias Zinc facility, treating 350,000 tonnes/year of zinc silicate concentrates. This process consisted of sulfuric acid leaching for zinc silicate concentrates and the traditional roasting-leaching-Zn electrowinning for zinc sulfide concentrates. Over 98% zinc was recovered by this process that provides good setting and filtration properties of the leaching residue with a minimum silica gel formation. The fact that zinc silicate ores were treated by using sulfuric acid solution as leaching agent under pressure is also a feasible process because of that the extraction of zinc into leaching solution and the remove of silica from leaching solution become more efficient while having the good separation performance of solid and liquid [15]. Microwave-assisted leaching [21] and ultrasound-assisted leaching [22] have also potential application in extraction of metals such as zinc and lead. Microwave irradiation was applied to quick leach zinc from zinc silicate ore, and could effectively eliminate silica and iron from the leaching solution, and improve the liquid-solid separation performance [23]. The use of ultrasound was investigated for the extraction of zinc from zinc industrial residues [24]. Other acid-based leaching processes given in the literature [25], such as heap leaching, in-situ leaching, etc., are only briefly considered. Among the different processing options available, sulfuric acid pressure leaching as an environmentally friendly process has attracted considerable attention.

Despite many researches were reported on sulfuric acid leaching of zinc silicate ores at 293–368 K, work on the high temperature leaching of siliceous zinc ores is scanty. Sulfuric acid leaching of zinc silicate ores having 65.1% hemimorphite and 14.25% smithsonite under pressure was studied [15], but further study on the high temperature leaching of willemite were conducted. Willemite as one of main carriers of zinc is presented in a number of oxidized zinc deposits. It was earlier reported by Terry and Monhemius [11] that sulfuric acid leaching of willemite was found to be difficult in comparison with those of hemimorphite. It seems to suggest that the direct leaching of willemite with sulfuric acid solution may need a higher acid concentration and/or longer leaching time. This may lead to the situation that the impurities (such as Fe and SiO_2 etc.) present in the raw material readily enter solution with the zinc, bringing about a burden on the downstream purification of leaching solution.

In this study, taking into account the characteristics (such as shorter leaching time, high selective extraction and metal recovery etc.) of high temperature leaching under pressure [26–28], an attempt was made to develop a hydrometallurgical process for treating willemite under high temperature. The current objective is to study the selective leaching characteristic of willemite in sulfuric acid solution under high temperature. A discussion on the said process as an attempt of laboratory scale study for producing good separation property of solid-liquid or the minimum requirements of downstream purification was made. Said process would be technically viable provided that maximum zinc extraction and minimal iron and silica dissolution could be achieved.

2. Experimental

2.1. Materials

The chemical component of willemite sample obtained from southwest China was analyzed by chemical methods as indicated in the book [29]. The results are shown in Table 1.

Table 1. Chemical component of willemite concentrate.

Component	Zn	Fe	Pb	Cu	S	As	SiO_2	Al_2O_3	K_2O	MgO	CaO
wt.%	43.96	1.23	0.78	0.03	0.06	<0.1	38.9	0.91	1.06	0.27	1.22

The phases identified by XRD analysis in the sample were mainly willemite and quartz, as shown in Figure 1. The occurrence of zinc in the sample was studied using phase analysis as reported by Zhang [30]. The results are listed in Table 2.

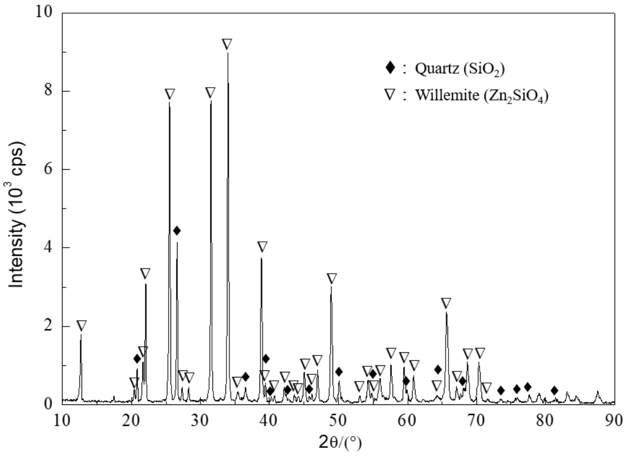

Figure 1. XRD pattern of willemite concentrate.

Table 2. The content and the phase distribution of zinc from willemite concentrate.

Constituent	Carbonate	Silicate	Sulfide	Others	Total
Zinc content (wt.%)	0.63	41.16	0.04	1.86	43.69
Zinc distribution ratio (%)	1.44	94.21	0.09	4.26	100

It was inferred from Figure 1 and Table 2 that willemite is the major zinc-containing phase in the sample. The scanning electron microscope coupled with energy dispersive X-ray spectrometer (SEM/EDS) was adopted to gain further knowledge of the mineralogical species and morphology of sample particles.

The results based on SEM/EDS analysis are shown in Figure 2. It can be seen that two distinct phases, quartz and willemite, with different colors and brightness were identified, with a composition in accordance with XRD results. The gangue mineral in the sample is mainly quartz. The solid particles present a smooth surface with a small number of pores and long fissures.

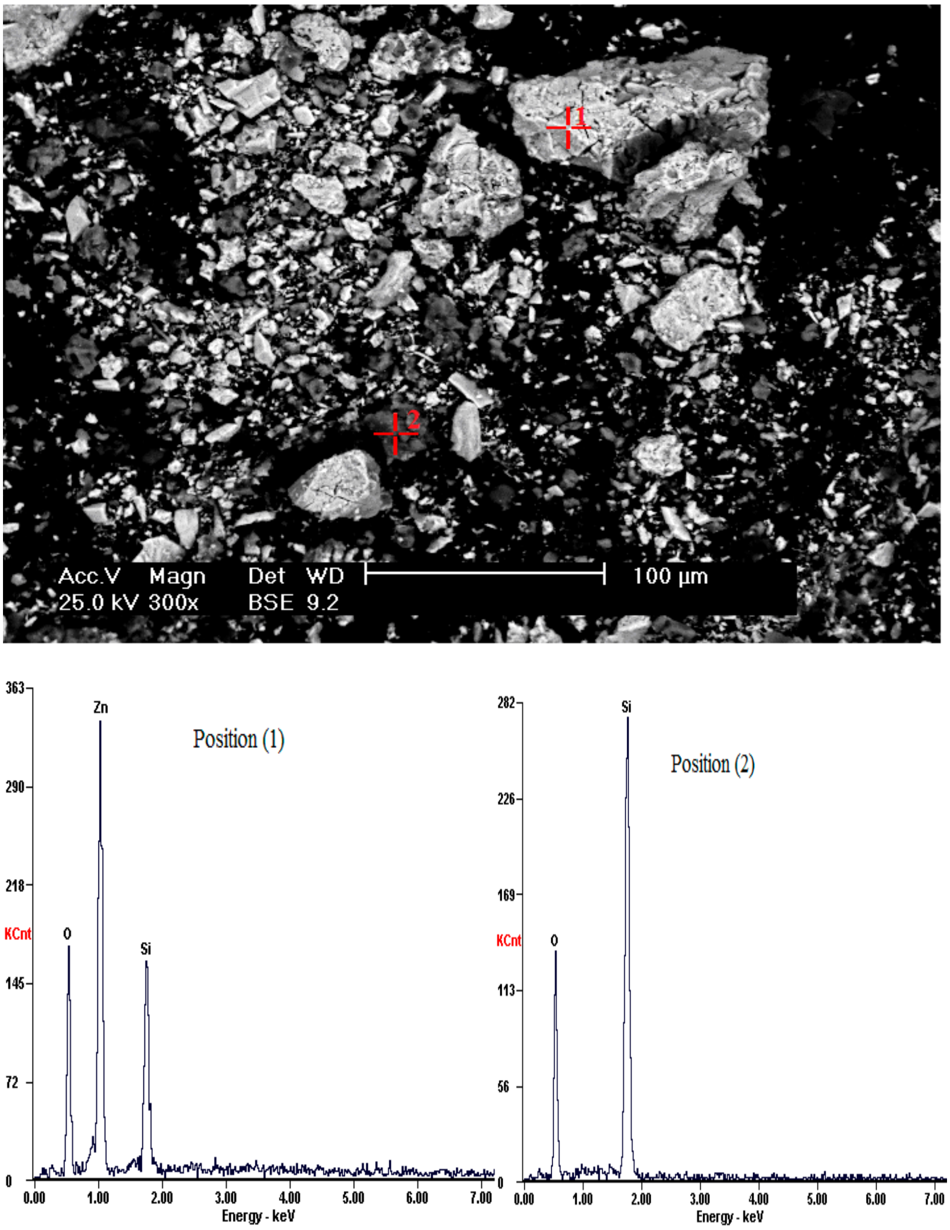

Figure 2. SEM image and EDS patterns of willemite concentrate (1-willemite, 2-quartz).

Analytically pure sulfuric acid (purity 98% and density 1.84 g/mL) was chosen as the leaching reagent in each run. Distilled water was used for preparation of all the aqueous solutions.

To study the affect of size distribution on dissolving efficiency, 1 kg of willemite concentrate was crushed, sieved and marked off several size fractions using Tyler standard sieve. The main analysis of each size fraction is shown in Table 3.

Table 3. Chemical analysis of each size fraction (wt.%).

Particle Size (Mesh)	Zn (%)	Fe (%)	SiO_2 (%)
−100 + 150	42.79	2.17	37.6
−150 + 200	43.35	1.43	38.4
−200 + 250	43.23	1.21	38.5
−250 + 300	43.96	1.23	38.9
−300 + 340	43.43	1.38	39.0

2.2. Equipment and Procedure

All the run were carried out in a 500 mL vertical titanium autoclave having a digitally controlled magnetic stirrer, an internal water-cooling titanium spiral tube and a gas vent through which oxygen was passed through solution. Stirring speed was obtained by a titanium impeller with three blades. Leaching temperature was adjusted with an accuracy of ±1 °C using a platinum resistance temperature sensor connected to a digital temperature controller.

In each case, 20 g willemite sample and 200 mL of sulfuric acid solution having the concentration specified were placed in the reaction chamber of the autoclave. The pulp was heated to the specified temperature in the 353–453 K range under oxygen pressure 1.2 MPa and stirring speed 550 rpm, unless otherwise specified. Each run was hold for specified duration, and then stopped heating, shut off pressure and cooled the samples to 343–353 K. The leaching slurry was removed from the reactor body and filtered. Leaching residues were washed twice using 20 mL sulfuric acid solution of pH 2.02 for recovering residual soluble zinc. The first filtrate and wash filtrate, measuring their volumes and pH, were sorted and collected. The residue dried fully was crushed and homogenized. Chemical analysis of leaching residue was carried out for the contents of zinc, iron and silicon.

3. Results and Discussion

3.1. Effect of H_2SO_4 Concentration

H_2SO_4 concentration (varying concentration in the 0.54–0.94 mol/L range) was studied in the first stage. The other conditions were as follows: L/S 10 mL/g, 413 K, particle size −250 + 300 mesh, agitation speed of 550 rpm, pressure of 1.2 MPa and 60 min duration. The results are shown in Figure 3, which shows that the extraction of zinc increases obviously with an increase in sulfuric acid concentration, and that 0.81 mol/L H_2SO_4 concentration is abundant in the over 97% zinc extraction within 60 min leaching time. Further zinc was not immediately available with higher concentrations of H_2SO_4. However, iron and silica dissolution increase slightly with increasing sulfuric acid concentration.

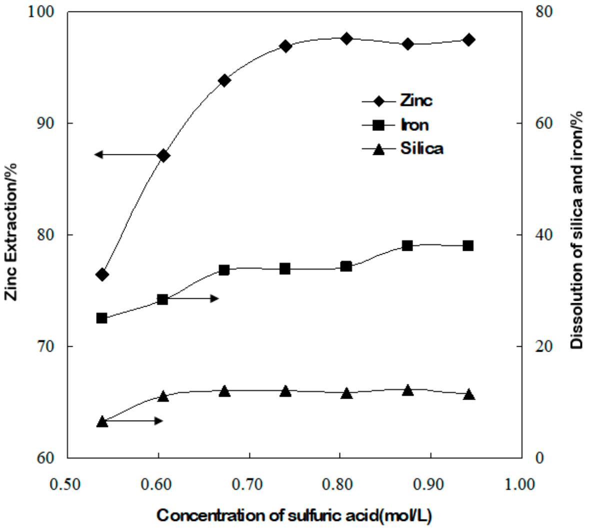

Figure 3. Effect of H_2SO_4 concentration on zinc extraction, and silica and iron dissolution.

Leaching behaviors of zinc in the present study resemble those of zinc silicate concentrates studied by Souza et al. [14]. Their materials contained mainly willemite, little hemimorphite and franklinite. In their study, zinc extraction was obviously affected by sulfuric acid concentration despite that the different experimental conditions were applied. To satisfy the solution purification requirements and fix minimum iron and silica dissolution problem, 0.81 mol/L H_2SO_4 concentration was considered the optimizing condition for the remaining experiments.

3.2. Effect of Temperature

For studying the optimization temperature, many tests were established with different leaching temperatures, while other ones were maintained as follows: 0.81 mol/L H_2SO_4 concentration, L/S 10 mL/g, particle size −250 + 300 mesh, agitation speed 550 rpm, 1.2 MPa pressure and leaching time of 60 min. Figure 4 shows the experimental results.

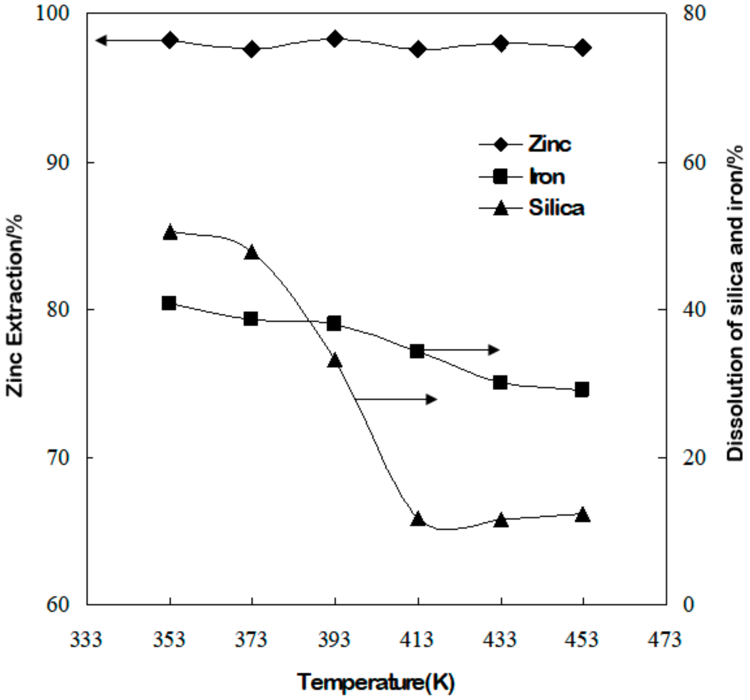

Figure 4. Effect of temperature on zinc extraction, and silica and iron dissolution.

It was inferred from Figure 4 that different temperatures used have no remarkable effect on zinc extraction but evidently affects iron and silica dissolution. The dissolution of silica decreases significantly with an increase in temperature stuedied, a stable period in performance is obtained after 413 K. The said behaviors could be because the formed silicic acid is decomposed. It was investigated by Iler [31] that under certain pressure, silicon sol was obtained from gel in the range of 373–573 K. However, the silicon sol behaviors could be changed under pressure leaching. This is because the direct dehydration of silicic acid was accelerated to form more filterable silica using the higher process temperatures. At higher temperature, the coagulation of silica sol could be promoted. Accordingly the dissolution of silica would be decreased. The iron dissolution is always in the tendency decrease under the used conditions. After 60 min, iron dissolution decreases from 40.85% at 353 K to 29.15% at 453 K. To obtain minimal iron and silica dissolution, 413 K would be more appropriate temperature.

3.3. Effect of Leaching Time

Each experiment on −250 + 300 mesh sample was carried out for studying the effect of duration under the conditions of 0.81 mol/L H_2SO_4 concentration, 413 K, L/S 10 mL/g, 550 rpm and pressure of 1.2 MPa. The results are plotted in Figure 5, which reveals that duration has remarkable influence on the dissolution of iron and silica but slightly affects the zinc extraction. As can be seen in Figure 5, as leaching time increases from 10 to 60 min, the dissolution of iron and silica decreases from 58.8 to 34.3%, and 45.3 to 11.8%, respectively, and iron and silica dissolution varies very little with a further increase in leaching time, while zinc extraction rises less than 3%. The said results resemble those of zinc silicate ores (hemimorphite as the main zinc-containing phase) obtained in our previous study. The remove of iron and silica increased with longer duration [32]. The silicic acid formed could be dehydrated entirely with longer duration. The more dehydrated silica would be brought into the final residue. Thus, leaching time for both lower energy consumption and minimal dissolution of silica and iron was standardized to 60 min.

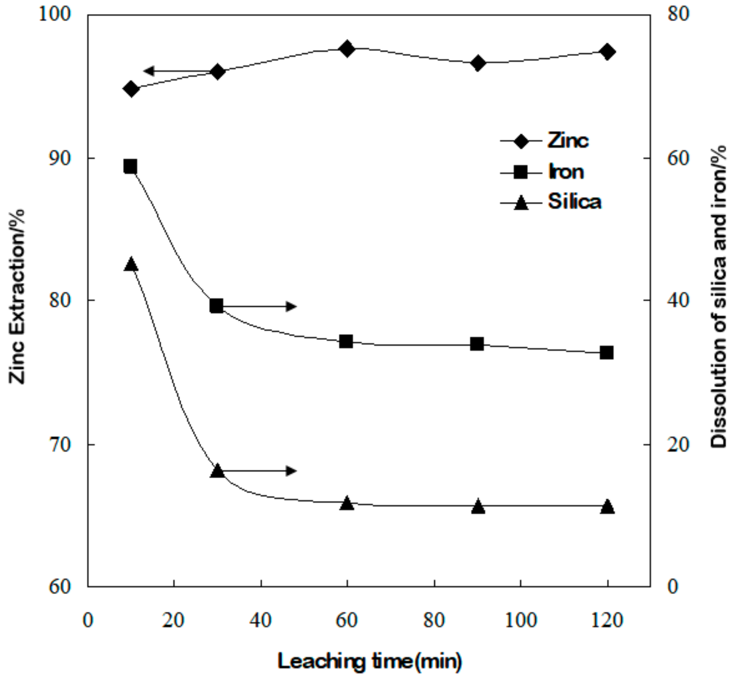

Figure 5. Effect of leaching time on zinc extraction, and silica and iron dissolution.

3.4. Effect of Liquid/Solid Ratio

The studied sample (−250 + 300 mesh) was leached in an agitated (550 rpm) solution containing constant acid addition (8.77 mL) within 60 min at 413 K and 1.2 MPa for different liquid/solid ratios in the range of 6–10 mL/g. The obtained results shown in Figure 6 reveal that liquid/solid ratio affects clearly iron and silica dissolution but zinc extraction slightly. Silica dissolution decreases from 27.2% at L/S 6 to 11.8% at L/S 10 with increasing of water content, and iron dissolution decreases slightly from 38.8% to 31.3%. In theory, silica dissolution should be improved with an increase of water content owing to the formation of silicic acid, as reported by Dufresne [9]. The said behaviors could be resulted from the silicic acid dehydration under the used conditions. In order to achieve minimal silica and iron dissolution, L/S ratio of 10 mL/g was chosen.

3.5. Effect of Pressure

Figure 7 shows the influence of pressure on willemite dissolution efficiency under the conditions of 0.81 mol/L H_2SO_4 concentration, L/S 10 mL/g, 413 K, agitation speed 550 rpm, particle size of −250 + 300 mesh and leaching time of 60 min. As shown in Figure 7, pressure has an influence on the dissolution of silica but appears to have a negative effect on both zinc leaching and iron dissolution. The silica dissolution, unchanged between 0.6 and 1.2 MPa, decreases from 11.8% at 1.2 MPa to 6.2% at 1.6 MPa. The said behaviors resembled those obtained from zinc silicate ores (hemimorphite as the main zinc-containing phase) by Xu et al. [32]. It was reported by Iler [31] that silica sol was obtained from gel through heating at high temperature of 373–573 K under pressure. The decrease in the extraction of silicon into the solution under high pressure and high temperature could be explained by the fact that the coagulation of silica sol could be promoted, and that the direct dehydration reaction of turning silicic acid into more filterable silica could be improved. Accordingly the silica dissolution would be decreased. Considering the safety factor of experiment and dissolution of silica, the optimal pressure was confirmed as 1.4 MPa.

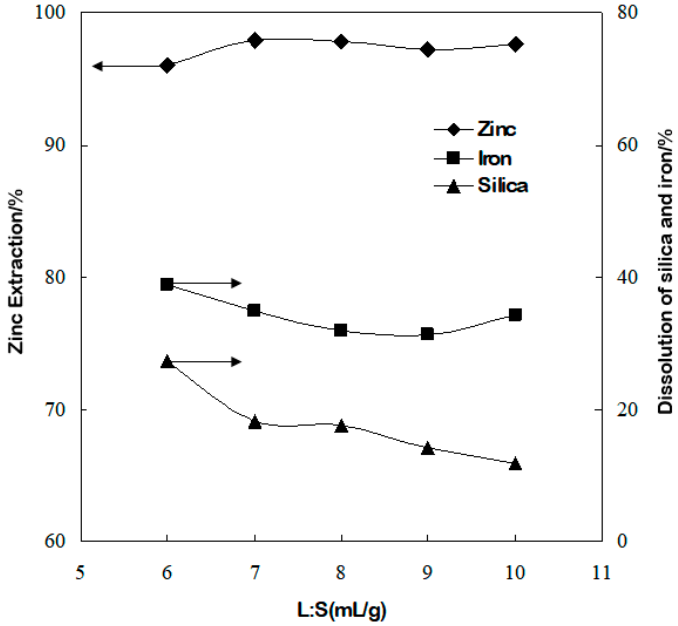

Figure 6. Effect of L/S on zinc extraction, and silica and iron dissolution.

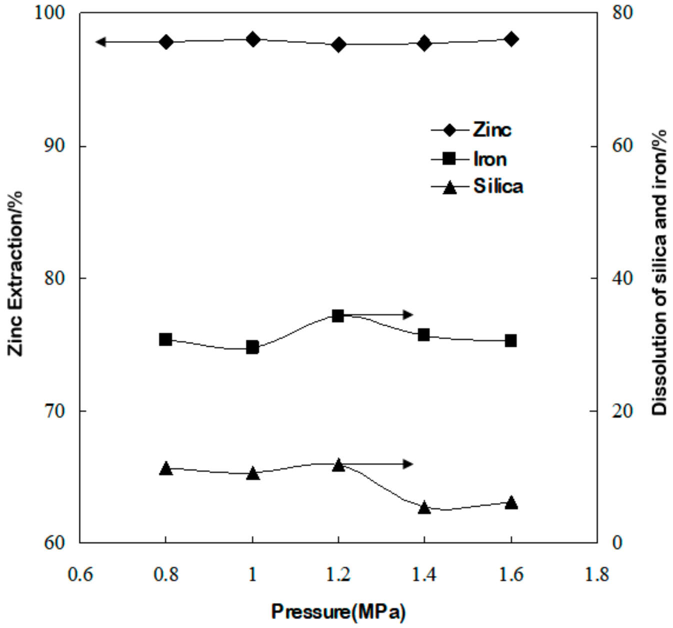

Figure 7. Effect of pressure on zinc extraction, and silica and iron dissolution.

3.6. Effect of Particle Size

Five different particle sizes (−100 + 150, −150 + 200, −200 + 250, −250 + 300 and −300 + 340 mesh) were used in the experiments with 0.81 mol/L sulfuric acid 413 K, L/S of 10 mL/g, 1.2 MPa, 550 rpm and leaching time of 60 min. From Figure 8, it is evident that the reaction rate is faster for the leaching of zinc and iron, while the particle sizes are smaller. This is because that decreasing the particle size will increase the contact area of particles with sulfuric acid. However, decreasing the particle size has no remarkable influence on silica dissolution. A particle size range of −250 + 300 mesh was applied in the rest of this study.

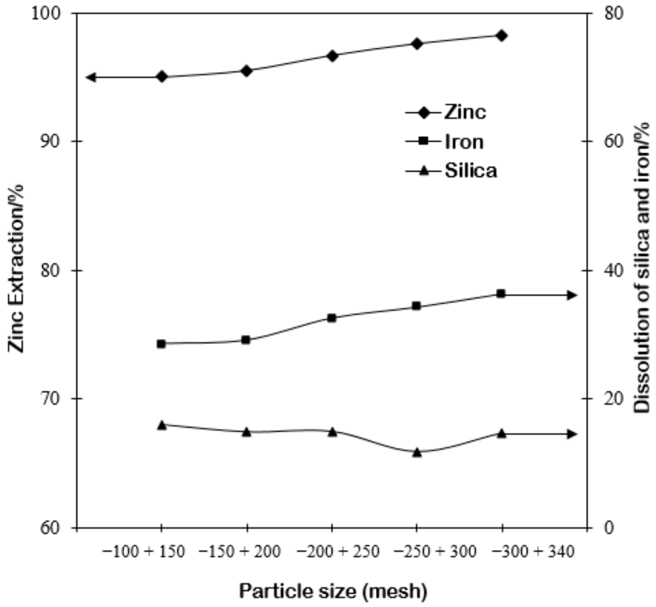

Figure 8. Effect of particle size on zinc extraction, and silica and iron dissolution.

3.7. Effect of Agitation Speed

The effect of stirring speed was investigated at various agitation speeds (250, 400, 550, 700, and 850 rpm) for 60 min at 413 K in solution containing 0.81 mol/L sulfuric acid and with L/S of 10 mL/g and particle size of −250 + 300 mesh.

Figure 9 shows that agitation speed has no significant effect on the dissolution efficiency of the studied sample. The dissolution values at 250 rpm are almost the same as those at 850 rpm. Therefore, in order to float solid particles and provide effective mixing in the lixivium, an agitation speed of 550 rpm was selected for investigating the effect of other parameters.

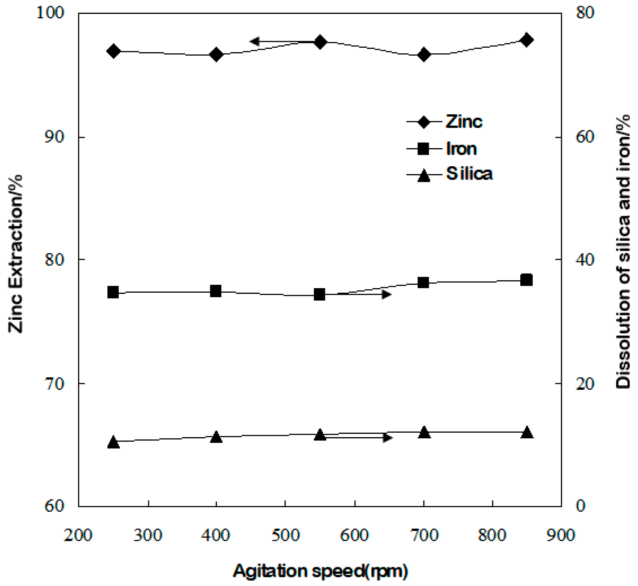

Figure 9. Effect of agitation speed on zinc extraction, and silica and iron dissolution.

3.8. Integrated Experiments

Through the above experiments, the optimal conditions were obtained as follows: 0.81 mol/L H_2SO_4, 1.4 MPa, 413 K, L/S of 10 mL/g, 550 rpm, leaching time of 60 min and particle size of −250 + 300 mesh. Under the optimal conditions, the verification experiments were carried out four times and the experimental results are plotted in Table 4. It was revealed that the leaching residue, having low lead, aluminum, zinc and other elements, contains mainly silica, iron and calcium.

Table 4. Experimental results under the optimum conditions.

Experiment No.		1	2	3	4	Average
Chemical composition of leaching residue (wt.%)	Zn	2.15	1.63	1.97	1.74	1.87
	Fe	2.13	1.89	2.06	1.95	2.01
	Pb	0.94	1.13	1.03	1.2	1.07
	As	<0.1	<0.1	<0.1	<0.1	<0.1
	SiO_2	77.04	75.6	76.3	74.69	75.9
	Al_2O_3	1.09	1.22	1.18	1.27	1.19
	K_2O	0.53	0.52	0.42	0.48	0.49
	MgO	0.2	0.22	0.23	0.22	0.22
	CaO	2.26	2.3	2.04	2.1	2.2
Leaching rate (%)	Zn	97.6	98.2	97.8	98.09	97.9
	Fe	15.58	27.01	18.35	23.51	21.11
	SiO_2	3.45	7.69	4.38	7.36	5.72

The leaching residue of experiment No. 2 was detected using XRD and SEM/EDS. The presence of quartz as the main phase of leaching residue was found by the XRD pattern (Figure 10). The SEM/EDS analysis is presented in Figure 11. It can be observed that the leaching residue identified contains mainly quartz, a small amount of franklinite and residual willemite, which are not found using XRD analysis owing to low content and their inhomogeneity. A scanning electron microscopy image, having a higher-resolution, enlarged view (Figure 11b), was shown at position 1 of Figure 11a. Through observation, the microstructure of residual willemite has a porous surface produced by sulfuric acid leaching process.

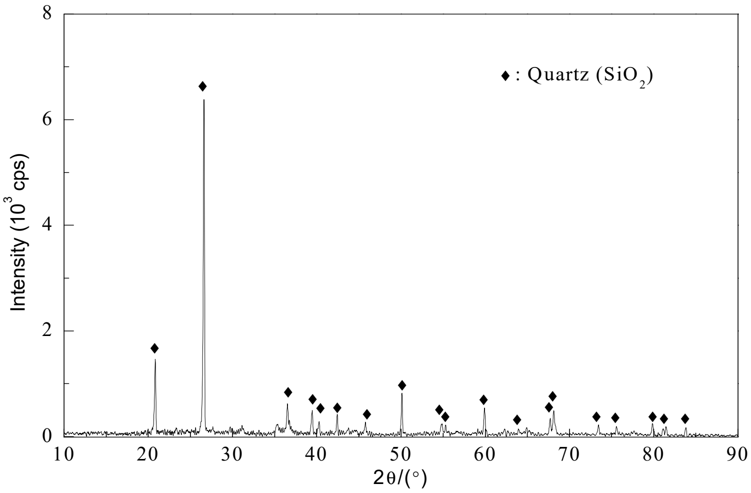

Figure 10. XRD pattern of leaching residue from the experiment No. 2.

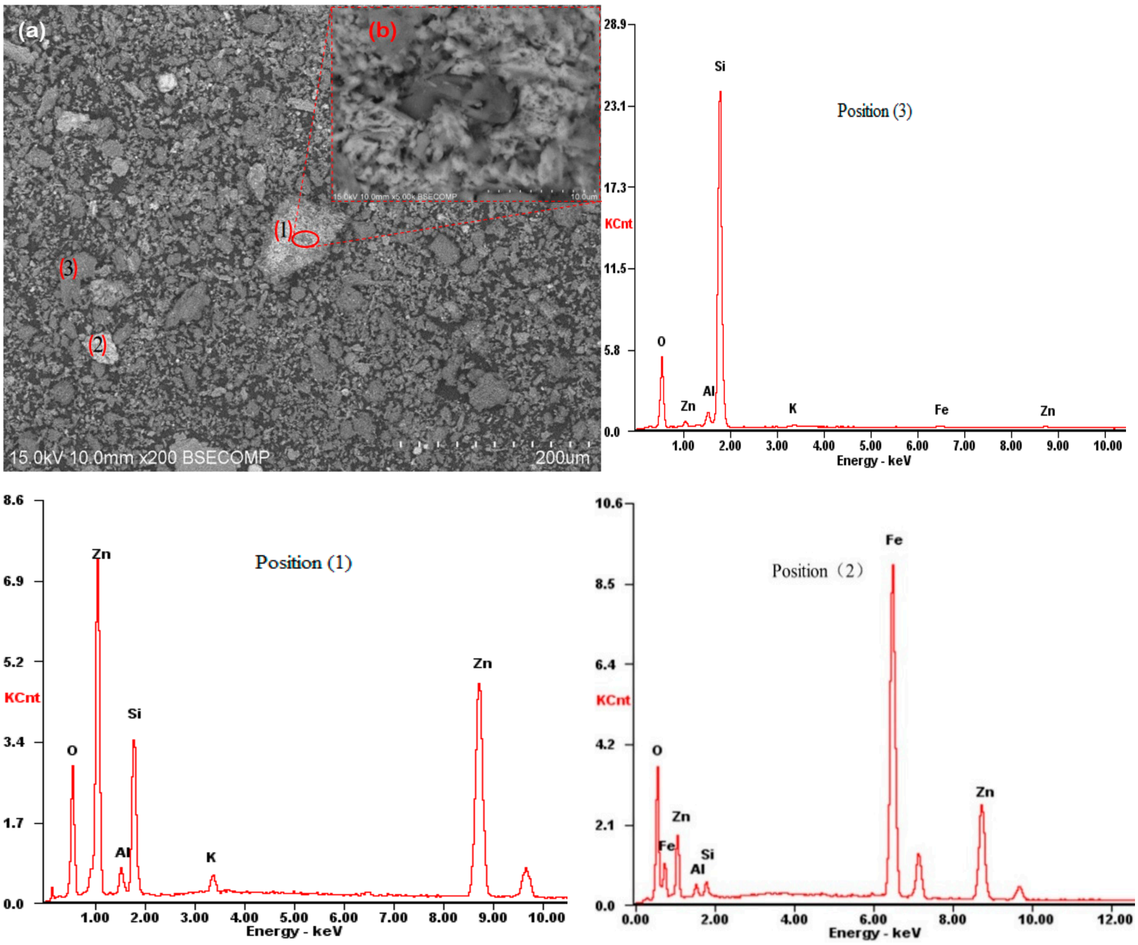

Figure 11. SEM images (**a**) ×200 times and (**b**) ×5000 times and EDS patterns of leaching residue from the experiment No. 2 ((1)-residual willemite, (2)-franklinite, (3)-quartz).

4. Conclusions

The technical feasibility of said process in the present study has been proven to be an attractive leaching process for treating willemite concentrate. The main conclusions were obtained as follows: (1) Under the optimum conditions (0.81 mol/L H_2SO_4, 413 K, 60 min leaching time, L/S of 10 mL/g, −250 + 300 mesh particle size, 550 rpm and 1.4 MPa pressure), over 97% Zn was extracted into the leaching solution with ca. 5.72% SiO_2 and 21.11% Fe. The minimum dissolution of impurities (such as iron, silica, and so on) had been achieved for lixivium purification when most zinc were leached from willemite sample. The separation performance of liquid-solid in the leaching slurry was good. (2) The most important parameter affecting the zinc extraction was sulfuric acid concentration, while the dissolution of iron and silica was affected most by leaching temperature. Zinc extraction increased significantly with increasing sulfuric acid concentration but iron and silica slightly. The iron and silica dissolution decreased gradually with increasing leaching temperature, while leaching temperature did not affect zinc extraction. The said behavior of silica dissolution was probably due to the fact that the formed silicic acid was broken down into more filterable silica. (3) It was inferred from the experimental analysis that three primary elements (zinc, iron and silicon) of willemite sample were changed into quartz and a small amount of franklinite.

Author Contributions: Conceptualization, H.X., Q.Z. and C.W.; data curation, H.X., Y.Q. M.Z. and J.X.; formal analysis, H.X. and Q.W.; investigation, H.X. and Y.Q.; methodology, Y.Q. and B.Z.; project administration, F.R.; resources, Q.Z. and C.W.; validation, H.X. and Y.Q.; visualization, W.Z. and F.T.; writing—original draft preparation, H.X.; writing—review and editing, Q.Z. and Q.W. All authors have read and agreed to the published version of the manuscript.

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