

Industrial symbiosis: High purity recovery of metals from Waelz sintering waste by aqueous SO₂ solution

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Abstract

Sintering operation in the production of Zn, Cd, and Pb by Waelz process produces a powdery waste containing mainly (about 70%) ZnO, CdO, and PbO. The waste may be referred to as *Waelz sintering waste* (WSW). The aim of this study is to develop a process for the separation and recovery of the metals from WSW with high purities. The process is based on the dissolution of the WSW in aqueous SO₂ solution. The research reported here concentrated on the effect of some important operational parameters on dissolution process. The parameters investigated and their ranges were as follows: SO₂ gas flow rate (*V*); 38–590 ml/min, stirring speed (*W*); 100–1000 rpm, reaction temperature (*T*); 13–60 °C, reaction time (*t*); 1–16 min, and solid–liquid ratio (*S/L*); 0.1–0.5 g/ml. The results showed that the dissolution rate increased with increasing *W*, *V*, and *S/L* and decreasing *T*. The best dissolution conditions were found to be *V* = 325 ml/min, *W* = 600 rpm, *t* = 6 min, *T* = 21 °C, and *S/L* = 0.1 g/ml. Separation of Zn from Cd involved precipitation of ZnSO₃ from a mixture solution. The best pH level for the precipitation was observed to be 6. © 2007 Elsevier B.V. All rights reserved.

Keywords: Waelz process; Industrial symbiosis; Leaching; Sulphur dioxide; Zinc sulphite

1. Introduction

Waelz process is a well-known metallurgical process in which zinc (Zn) ore or concentrate is heated with fuel oil, coke or powdered coal in a reducing rotary kiln, known as Waelz Furnace, at 1100–1200 °C [1]. The Zn and other volatile non-ferrous metals in the feed are vaporized in the furnace and carried to an external collection system comprising a cyclone and baghouse. The collected dust, called as waelz oxide, is a crude Zn bearing product. The waelz oxide is further refined by a second kiln step where the material is further heated and sintered to form a Zn clinker material [2].

At the sintering stage in the production of Zn, cadmium (Cd), and lead (Pb), where harmful components for the electrolysis such as F and Cl are removed, a powdery waste product is obtained from the condensation of metal vapours. This waste material, which we name as “Waelz sintering waste” (WSW) in

this article, contains mainly (up to 70%) ZnO, CdO, and PbO. Currently there is no route for the utilisation of this valuable material due to unavailability of suitable technologies for the separation and recovery of the metals with high purity.

Zn is used in many materials that find applications from automotive industry to galvanization. Materials containing Cd also finds usage in many applications such as electro-plating, Ni–Cd batteries, standard emf cells, control of atomic fusion, and paint industry. Pb is used in batteries, in X-ray equipments, and in nuclear power plants [3].

Development of possible routes for the utilisation of WSW may be inspired by the activities on the production of these metals (Zn, Cd, and Pb) from their minerals. With this in mind, here we will present a brief review on the production of these metals from their minerals. The wealth of information and knowledge that is already available in the relevant field can be called upon while developing possible routes for the utilisation of WSW.

Goldstein et al. [4] produced ZnSO₄·7H₂O with 99.5% purity from oxide minerals with high content of Zn and iron (Fe) by H₂SO₄ leaching. Zapuskalova et al. [5] investigated the selectivity of the acidity in acid leaching stage of a Zn sintering process.

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Dimanche et al. [6] investigated the leaching of zinc oxide minerals in H_2SO_4 , H_2SO_3 , NaOH, and NH_4OH . They reported that in the leaching of the samples that were rich in iron oxide NaOH was more effective than H_2SO_4 and that the effect of NH_4OH was almost insignificant. Salikhov et al. [7] produced Zn and other leachable metals with high purities from solutions by minimizing the contamination of the solution with impurities like Fe, Al, Sb, Ge, and As. Frenay [8] studied the leaching of zinc oxide minerals in different media. It was reported that at 20 °C H_2SO_4 and NaOH gave better results than NH_4OH . Shakhtaktinski et al. [9] have studied the leaching of roasted minerals and reported that the best results were obtained for 200 g H_2SO_4/l , 90 °C, 120 min and 1/10 solid–liquid ratio. Rbouche and Kerdoudj [10] have studied the production of Zn from Pb–Zn oxide minerals. They found that the highest production rate was obtained from a leaching process of 3 h with 0.5 M HCl after roasting the mineral for 0.5 h at 400 °C.

Sulphur dioxide (SO_2) is generated, often as a harmful waste, in large quantities by various industrial processes changing from oil and gas operations to roasting processes of ores with sulphide content. SO_2 is very harmful to environment and health and difficult to handle due to its hazardous nature. Depending on the quality, quantity, and location, SO_2 may be utilised and converted to valuable products. The largest demand for SO_2 is from the production of sulphuric acid (H_2SO_4). SO_2 also finds usage in hydrometallurgy as a leaching agent and this is the characteristics of SO_2 that we believe might be exploited in the recovery of metals from WSW.

Sevilla et al. [11] determined the solubility of dilute SO_2 in aqueous HCl and NaCl solutions at 298 K as a function of ionic strength, up to 3 mol/l, and the partial pressure in the range between 0.075 and 1.8 kPa. They proposed an equation to correlate the apparent Henry's law constant and the apparent first dissociation constant as a function of ionic strength. Gbor et al. [12] investigated the effectiveness of aqueous SO_2 in extracting metals from discarded smelter slag and assessed the dissolution behaviour of Co, Ni, and Fe using aqueous SO_2 and H_2SO_4 . They suggested that both acid and sulphur(IV) ligand attacked the reactive sites on the smelter slag surface to dissolve the metals. The aqueous SO_2 solution was found to be more effective in leaching than H_2SO_4 at the same pH. The effect of acid was more predominant at lower pH, while the ligand effect of aqueous SO_2 became stronger at higher pH. Voegelé et al. [13] found that starting from hydrated SO_2 the conversion into HSO_3^- did not take place directly. They suggested that the mechanism of HSO_3^- formation was a two-step process: first, SO_2 is hydrated to form bisulphite SO_2OH^- and then SO_2OH^- is converted sulphionate ion HSO_3^- .

The composition of the material used in the present study is given in Table 1. As seen its main constituents are ZnO, CdO, and PbO. The aim by the present study is to help to develop a

process for the recovery of valuable metals from WSW. A possible processing scheme is depicted schematically in Fig. 1. The process follows a route that first separates Zn and Cd from Pb by dissolving ZnO and CdO in aqueous SO_2 solutions. It then produces a mixture of zinc sulphite ($ZnSO_3$) and cadmium sulphite ($CdSO_3$) from the acidic Zn and Cd solution. Later $ZnSO_3$ is crystallised from this solution in the form of various hydrates, $ZnSO_3 \cdot nH_2O$, with $n = 3, 5/2, 2$ and 1, depending on the temperature, pH, and composition of the solution, especially the amount of the sulphurous acid. $CdSO_3$ also crystallizes from aqueous solutions either as hydrates or in the form of the anhydrous salt [14] but with significantly lesser amount. $ZnSO_3$ is mainly used for medical purposes [3]. To the knowledge of the authors there has been no study done on the production of $ZnSO_3$ from Zn containing minerals.

The present study proposes a process for the recovery Pb, Zn, and Cd from a waste product (WSW) by making use of an environmentally harmful gas (SO_2). We believe that this study will contribute to waste minimisation, wealth and job creation, environmental impact abatement, and sustainable development. We, therefore, hope that the work presented here will be of the value to the academic and industrial circles with activities in industrial symbiosis.

2. Materials and methods

The mineral material, WSW, used in this investigation was provided by ÇINKUR in Kayseri province, Turkey, which produces Zn, Cd, and Pb. As WSW was already in powder form, there was no need for crushing, grinding, and sifting. WSW as collected from the production site in different bags was first blended thoroughly before dividing and re-packing it into 20–25 kg sample bags. The particle size was found to be below 74 μm by conventional sieve analysis. Chemical analysis of the WSW was performed by standard volumetric methods and a SHIMADZU 670 atomic absorption spectrophotometer. The average composition is given in Table 1.

The dissolution operation was carried out in a spherical glass reactor of 250 ml volume fitted with a mechanical stirrer, gas sparger system, and a refluxed condenser opened to atmosphere. Atmospheric condition in the lab was, on average, 610 mmHg and 21 °C. The reactor was placed in a temperature controlled water bath. Water of 100 ml volume was taken into the reactor and saturated by SO_2 gas with a fixed flow rate through the sparging system while stirring the content with a predetermined speed. Once the desired temperature reached, an amount of WSW sample to give a certain S/L ratio was quickly added into the reactor. SO_2 gas flow through the reactor was continuous and kept constant throughout the reaction period. Immediately after the end of the reaction period, the content of the reactor was filtered at the room temperature. The filtrate then analyzed

Table 1
The composition of Waelz sintering waste used in the dissolution tests

Element	Zn	Cd	Pb	Cu	Mg	Ni	Sb	Co	Mn	Fe ₂ O ₃	CaO	Cl ⁻	SO ₄ ²⁻
%	17.65	5.58	42.85	0.013	0.013	0.012	0.038	0.008	0.025	0.13	0.014	5.55	2.28

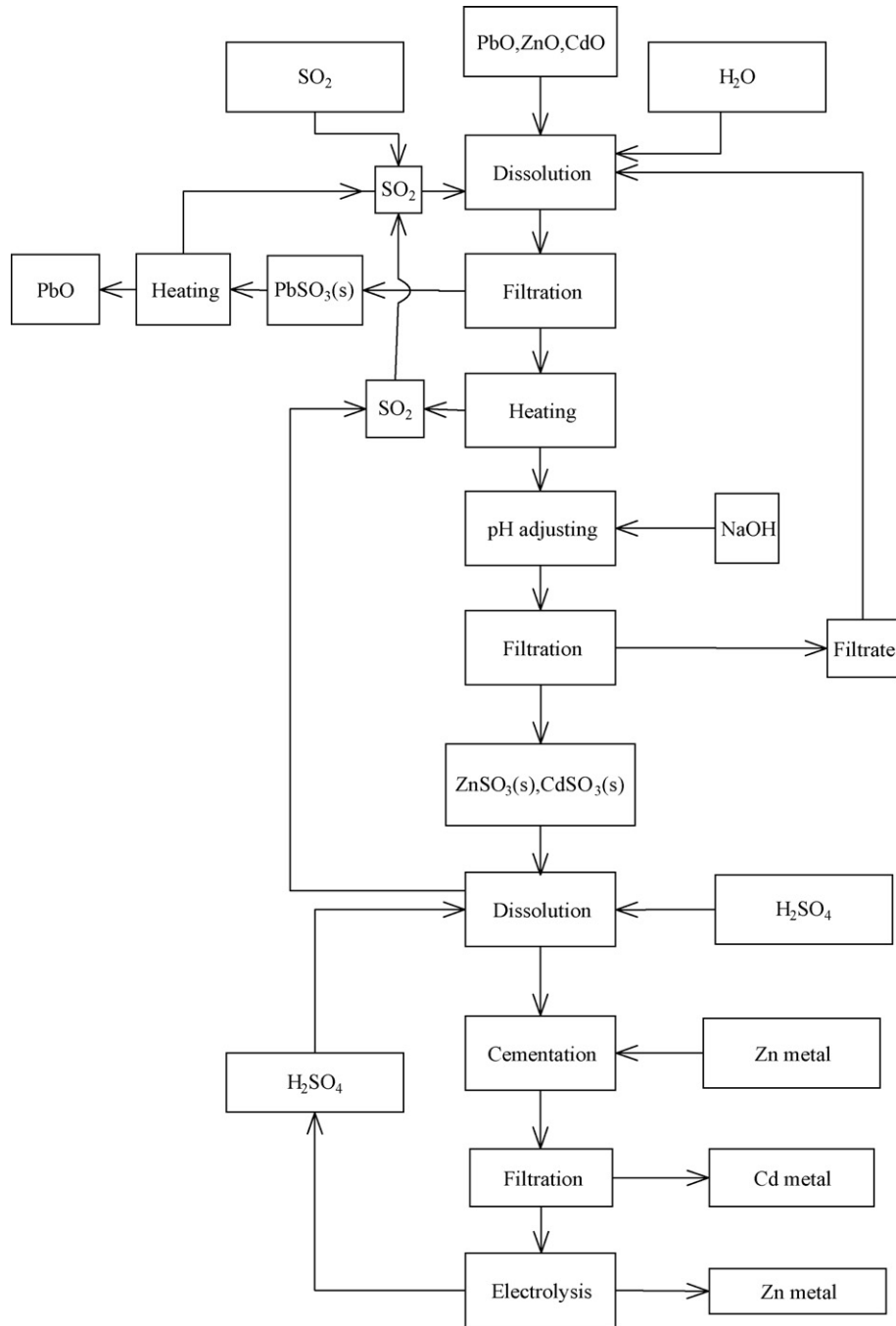


Fig. 1. A simplified block diagram for the production of Zn and Cd through the dissolution of Waelz sintering waste in aqueous SO_2 solutions.

for Cd, Pb, and Zn content. The dissolved quantities of Zn and Cd were first determined together by complexometric method using Titrplex III as complexing agent. Later Cd content was determined separately by atomic absorption spectrophotometer thus enabling Zn contentment to be calculated from the total Zn and Cd analysis.

3. Results and discussions

The experimental work concentrated on the dissolution of WSW in aqueous SO_2 solution and precipitation of ZnSO_3

from the solution by pH adjustment as function of the important operational parameters. These were SO_2 gas flow rate (V), stirring speed (W), reaction temperature (T), reaction time (t), and solid–liquid ratio (S/L). In what follows, the dissolution term refers to the total dissolution percentage of Zn and Cd.

3.1. The effect of SO_2 flow rate

The effect of SO_2 flow rate (V) on the dissolution was investigated in the range between 38 and 590 ml/min. The other parameters were kept constant at: $t = 5$ min, $W = 500$ rpm,

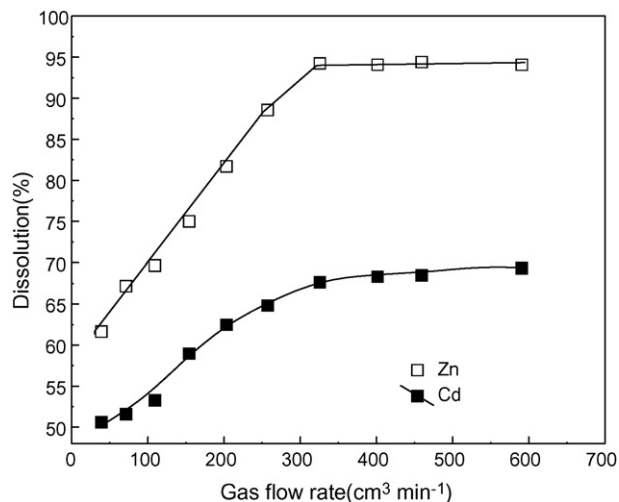


Fig. 2. The effect of the gas flow rate on the dissolution. $W = 500$ rpm, $T = 25$ °C, $t = 5$ min $S/L = 1/3$.

$T = 25$ °C, and $S/L = 0.333$ g/ml. The results obtained are shown in Fig. 2. The dissolution first increases rapidly with V up to 325 ml/min and thereafter it remains constant. This means that at low V values SO_2 absorption is perhaps not complete leading to SO_2 deficiency in the solution and thus reduction in the dissolution of WSW. The best V may be taken as 325 ml/min since further increase in V does not significantly influences the dissolution. Therefore, when investigating the effect of other parameters, V was kept constant at 325 ml/min. It was observed that $PbSO_3$ did not dissolve remarkably in SO_2 solutions.

3.2. The effect of reaction time

The effect of the time (t) on the dissolution is shown in Fig. 3. The values of the other parameters were same as those in the effect of V . As seen from this figure, the dissolution increases with t up to approximately 5 min beyond which no considerable increase in the dissolution is observed. Thus the best t has been

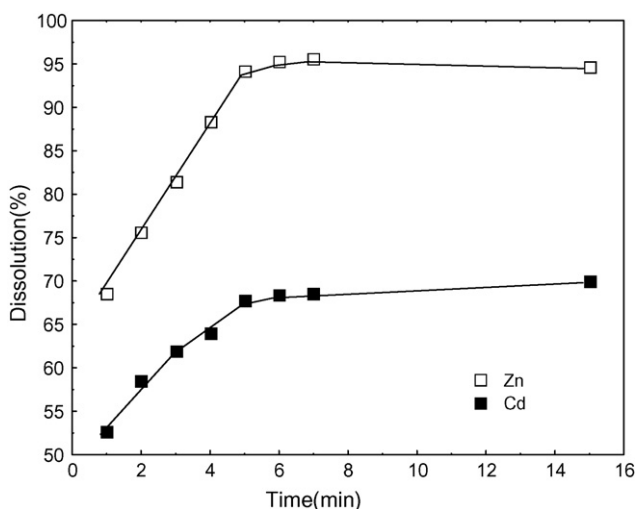


Fig. 3. The effect of the time on the dissolution. $W = 500$ rpm, $T = 25$ °C, $V = 325$ ml/min, $S/L = 1/3$.

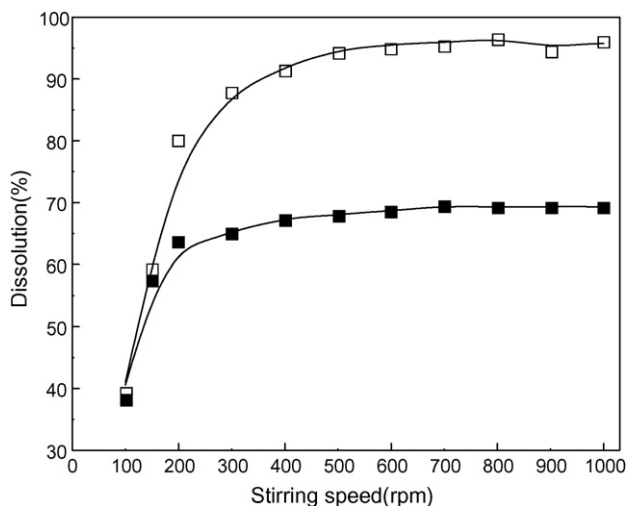


Fig. 4. The effect of the stirring speed on the dissolution. $t = 5$ min, $T = 25$ °C, $V = 325$ ml/min $S/L = 1/3$.

taken as 5 min and when investigating the effect of the other parameters, t was kept constant at this value.

3.3. The effect of stirring speed

The effect of stirring speed (W) on the dissolution was investigated in the range 100–1000 rpm. S/L and T were kept constant at 0.333 g/ml and 25 °C, respectively. The results obtained are shown in Fig. 4. As seen from this figure the dissolution first increases significantly with increasing W up to 400 rpm, it then increases slightly between 400 and 600 rpm, and thereafter it remains constant. This indicates that the reaction is probably film layer mass transfer controlled. The stirring is expected to reduce the liquid film thickness surrounding solid particles, through which the gas is transferred to the solid surface, as well as enhance the renewal of the film thus leading to increased mass transfer from the bulk liquid phase to the interface. The dissolution of solids by gaseous solutions are often heterogeneous in character and takes place through four sequential steps [15]: (1) the transfer of the gaseous reactant from the bulk gas phase into the solution (the dissolution of gas in the liquid); (2) the diffusion of the reactant from the solution onto the S/L interface through the interfacial film; (3) the reaction of the reactant (gas) with the solid at the surface and, if porous, in the pores as well; (4) the diffusion of the products from the interface into the solution. The mass transfer resistances of these steps normally differ from each other and the one with the highest resistance controls the reaction rate. For highly soluble gases with fast reaction kinetics, the dominant resistance is due to the diffusional mass transfer through the liquid film. The resistance in the film, for a given solid–gas–liquid system, is dependent on the diffusion coefficient and film thickness, which in turn are functions of the system thermodynamic properties (pressure and temperature) and the fluid dynamics conditions. The film thickness decreases with the increases in the relative velocity of the liquid over the solid surface [16]. Under these circumstances, the mass transfer coefficient is a function of Reynolds number (Re) and the mass transfer increases with the increases in the stirring intensity. The

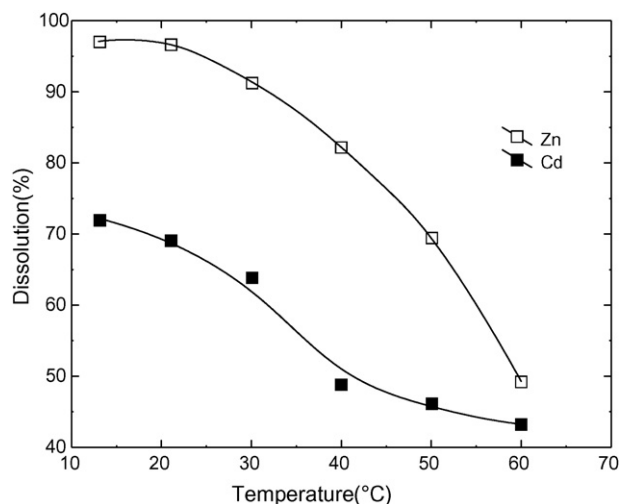


Fig. 5. The effect of the temperature on the dissolution. $W=500$ rpm, $V=325$ ml/min, $t=5$ min $S/L=1/3$.

increase in the dissolution with the increase in W displayed by the present data under investigated conditions, thus, indicates that the dissolution process in the question is controlled by the interfacial diffusion that in turn is determined mainly by the film thickness and film renewal rather than changes in the diffusion coefficient as discussed below in the section on the effect of temperature.

W of 600 rpm has been taken as the best stirring speed and when investigating the effect of the other parameters, W was kept constant at 600 rpm.

3.4. The effect of temperature

The effect of the temperature (T) on the dissolution was investigated in the range 13–60 °C. The results obtained are shown in Fig. 5. It can be seen from this figure that the dissolution decreases with increasing T . This negative temperature dependency indicates that, under investigated process conditions, the process is neither diffusion coefficient nor chemical reaction kinetics controlled. Because if it was, the dissolution would be increased with the temperature as both the kinetics (Arrhenius model for reaction rate constants) and diffusion (Arrhenius model for diffusion coefficients) would be improved with the temperature. The behaviour displayed by the data presented in Fig. 5 can be explained by the reduction in the solubility of SO_2 with increasing T under constant pressure. Under atmospheric pressure, the solubility of SO_2 in water is 18.59 and 8.60% at 0 and 25 °C, respectively [17]. As the temperature increases dissolved SO_2 concentration in the solution becomes insufficient resulting in the decreases in the dissolution of the WSW. That the dissolution decreases with the temperature and increases with the stirring speed is a further indication that the dissolution reaction in the question is controlled by the thickness of the interfacial film and its renewal rate as result of the increased turbulence with stirring. The dissolution appears to be controlled by the fluid dynamics of the system.

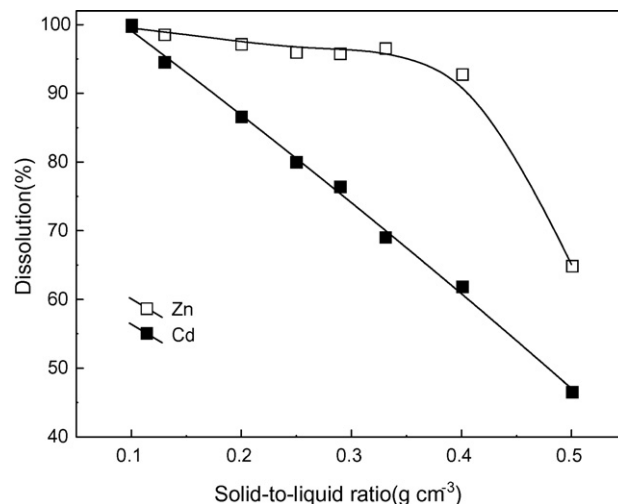


Fig. 6. The effect of S/L ratio on the dissolution $W=500$ rpm, $T=25$ °C, $t=5$ min, $V=325$ ml/min.

As it is seen from Fig. 5, the best dissolution is obtained at 13 °C. But because this would require cooling and the fact that the dissolution does not differ significantly from 13 °C to the laboratory temperature (21 °C), when investigating the effect of the following parameter (S/L), T was kept constant at 21 °C.

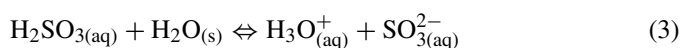
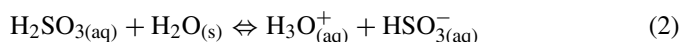
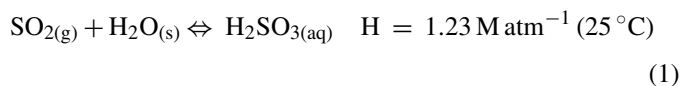
3.5. The effect of S/L ratio

The effect of the S/L ratio on the dissolution rate was investigated in the range of 0.1–0.5 g/ml. The results obtained are shown in Fig. 6. An increase in the dissolution is observed with decreasing S/L . For an S/L of 0.1 g/ml the dissolution of Zn and Cd are 99.81 and 100%, respectively.

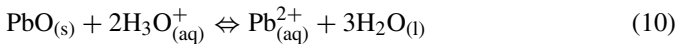
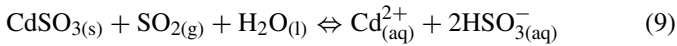
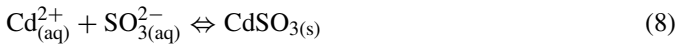
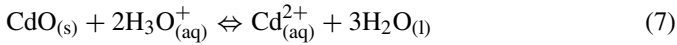
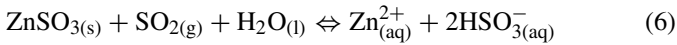
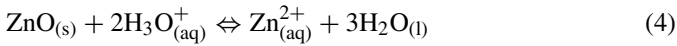
In the view of the above results the best dissolution conditions were found to be $V=325$ ml/min, $W=600$ rpm, $t=5$ min, $T=21$ °C and $S/L=0.1$ g/ml.

3.6. Mechanism of dissolution reactions

Sulphur dioxide easily dissolves in water to form H_2SO_3 . The solubilisation takes place through formation of two salts; first bisulphite (HSO_3^-) and then sulphite (SO_3^{2-}). The pH of the solution varies depending on the equilibrium between HSO_3^- and SO_3^{2-} . Distribution of sulphur(IV) species at various pH has been investigated by Gbor et al. [18]. According to the data reported, ionised SO_2 is the dominant specie for pH smaller than 1.5, HSO_3^- for pH between 1.5 and 6.5, and SO_3^{2-} for pH above 6.5. For pH values of less than 2, ionised SO_2 and HSO_3^- form the total sulphur(IV). The chemical reactions during the dissolution of SO_2 in water are as follows [12]:



and the chemical reactions of the WSW in aqueous SO₂ solution are:



According to chemical reactions (1)–(3), when the SO₂ concentration increases in the medium, the pH will be lowered and the equilibrium will progress towards the right. At the same time the concentration of Zn²⁺, Cd²⁺, and HSO₃⁻ will rise as result of the dissolution of ZnSO₃ and CdSO₃.

Lutz and Engelen [14] reported that the solubility of ZnSO₃ increases with SO₂ concentration. There seems to be no information in the literature for the solubility of CdSO₃ with SO₂. But from the data presented in Figs. 2 and 3, where it is observed that the solubility increases with both SO₂ flow rate and reaction time, it is deduced that the dissolution of ZnSO₃ as well as that of CdSO₃ must be increasing with the total SO₂ composition. We believe that the incomplete dissolution of CdO and ZnO displayed by some of the results reported above are due limited SO₂ concentration (absorption) in the leaching solution. It has been shown elsewhere [14] that the aqueous solubility of ZnSO₃ increases with medium temperature whereas that of CdSO₃ decreases. But the data from the present work given in Fig. 5 indicate that the solubility of both ZnSO₃ and CdSO₃ decreases with the temperature. Considering the behaviour displayed in Fig. 5 together with the chemical reactions (6) and (9), we believe that this is because the effect of H₂SO₃ concentration on the dissolution ZnSO₃ and CdSO₃ is more dominant than that of the medium temperature.

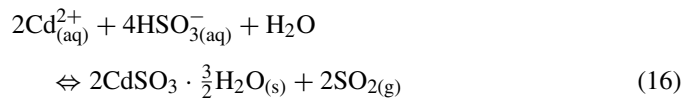
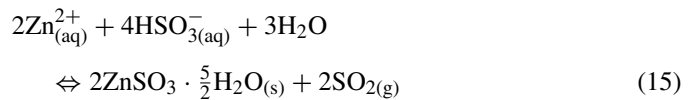
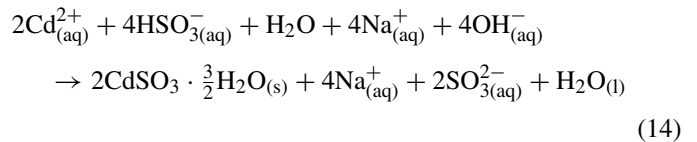
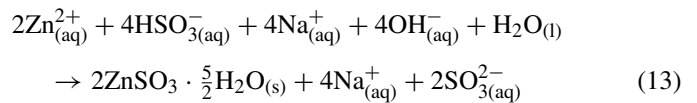
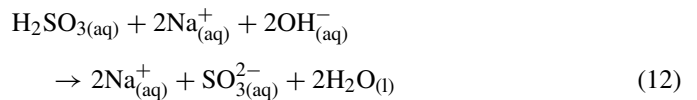
3.7. Separation of Zn from Cd

It was observed in the present study that PbSO₃ does not dissolve remarkably in SO₂ solutions confirming earlier studies reported elsewhere [19] that PbSO₃ is insoluble in water. The method proposed in the present study on the separation and recovery of metals from WSW is based on this character of PbSO₃ and the separation of Zn and Cd by dissolution. The solubility of zinc sulphite (ZnSO₃) in water at 20 °C is 1 × 10⁻² mol ZnSO₃/kg H₂O whereas that of cadmium sulphite (CdSO₃) is 2.21 × 10⁻³ mol CdSO₃/kg H₂O [14]. As the solubility difference is more than one magnitude of order, Zn and Cd can easily be separated from the solution by precipitation. A possible block diagram representation of the proposed route of processing is shown in Fig. 1.

Larsen and Linkson [20] developed the potential diagram of the Zn–SO₂–H₂O system at 25 °C for a total sulphite activity of 1.0 mol/l. According to this data, at pH 5, a solution of 10⁻³ M active Zn concentration is in equilibrium with ZnSO₃·2.5H₂O. It should be noted that the precipitation of ZnSO₃·2.5H₂O is realised at lower pH values than the pH values for the precipitation of zinc hydroxide, Zn(OH)₂. ZnSO₃ is a white crystalline material with easier filterability than Zn(OH)₂.

Following the solubilisation of the metals, Cd and Zn, in aqueous SO₂ solution, the precipitation of ZnSO₃·nH₂O from the solution was carried out by adjusting the pH of the solution to 6 by the addition of NaOH. The precipitate was filtered from the solution and dried. Lutz and Engelen [14] reports that the stable hydrate of ZnSO₃ at ambient temperature is in the form of ZnSO₃·2.5H₂O. This was confirmed by the X-ray analysis of the precipitates obtained in the present work.

The reactions taking place in the precipitation process are thought be as follows:



The solubility of ZnSO₃·2.5 H₂O in water at ambient conditions is approximately 1.5 g ZnSO₃/kg H₂O and it decreases by the presence of Na₂SO₃ up to 1.1 mass % Na₂SO₃ and increases at higher concentrations of Na₂SO₃ [14]. Under the current experimental conditions (atmospheric pressure of 610 mmHg and a solubility of SO₂ of 1.2 mol/l) the presence of Na₂SO₃ was estimated to be maximum 13 mass %. Considering the findings reported by Lutz and Engelen [14], it was calculated that, in the presence of this amount of Na₂SO₃, the solubility of ZnSO₃ is maximum 0.73 mass %. In the best dissolution conditions (S/L = 0.1 g/ml) this is equivalent to 20 mass % of available ZnSO₃ in the solution. This, thus, implies that over 80 mass % of the all CdSO₃ and ZnSO₃ has been precipitated. If the process was to be carried out with an S/L value of 0.3 or higher, over 90 mass % of the total dissolved CdSO₃ and ZnSO₃ could have been precipitated.

4. Conclusions

This paper aims to develop a possible processing route for the recovery of valuable metals (Zn, Cd, and Pb) with high purities from Waelz sintering waste (solid) through first partial solubilisation in aqueous SO₂ solutions and then precipitations by adjusting the pH of the solution. Effect of some important operational parameters (gas flow rate (*V*), stirring speed (*W*), reaction temperature (*T*), reaction time (*t*), and solid-to-liquid ratio (*S/L*)) were studied. The results confirmed the possibility of a processing route for the metal recovery from WSW through industrial symbiosis and lead to following conclusions:

- It possible to recover Zn, Cd, and Pb separately and with high purities from Waelz sintering waste through a simple process involving mainly solubilisation of the waste solid by another essentially waste material, aqueous SO₂ solution, and precipitation by pH adjustment.
- The dissolution process is controlled by the interfacial diffusion that in turn is determined mainly by the film thickness and film renewal rate rather than changes in the diffusion coefficient. That is the process fluid dynamics control the dissolution.
- The dissolution increases with increasing SO₂ flow rate and stirring speed but decreasing S/L ratio and temperature.
- The fact that Pb of Waelz sintering waste powder is not dissolved at all but its Cd and Zn are dissolved 100% in aqueous SO₂ solutions suggests that SO₂ may be used as a selective leaching agent.
- For a 100% leaching efficiency in laboratory conditions, the most suitable conditions have been found to be as follows: gas flow rate (ml/min): 325; reaction time (min): 5; stirring speed (rpm): 600; temperature (°C): 21; S/L ratio (g/ml): 0.1
- It is quite possible that a leaching efficiency of 100% may also be obtained under different operating conditions than those most suitable conditions observed in this experimental work. Which set of the processing conditions will be better from economical and technical point of view can be assessed by a detailed optimisation study and carrying out some confirmation tests, preferably at pilot plant scale.
- ZnSO₃·5/2H₂O and CdSO₃·3/2H₂O can be separated out by precipitation from the leaching solution by adjusting the pH of the solution to near neutral (pH 6). The precipitate is mainly ZnSO₃·5/2H₂O due to significantly higher solubility of CdSO₃·3/2H₂O enabling the relatively high purity separation of Zn and Cd.

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