Structural Destruction of As–Sb Solid Solution through a Selective Oxidation Process in the Presence of CaO and Its Effect on As Removal from the As–Sb Dust

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ABSTRACT: As–Sb dust from metallurgical processes causes serious environmental issues due to the presence of the toxic element As. In this study, a facile method for removing As through the destruction of the As–Sb solid solution structure from the dust using CaO under an oxidizing atmosphere was processed. The effects of the variables, including the amount of CaO added, roasting temperature and time, and oxygen partial pressure, on the volatilization of As and Sb were investigated. Under an oxidizing atmosphere in the presence of CaO, the formation of the vitreous material (As–Sb−O) was hindered through the transformation of Sb2O3 and (As, Sb)2O3 in the untreated dust into Sb2O4, Sb2O5, Sb6O13, and Ca2Sb2O7, and the volatilization of arsenic was promoted, as a result. The volatilization attained for As was 91.53% whereas that for Sb was only 4.30% under the following optimum conditions: 5 mass % CaO, a roasting temperature of 550 °C, an O2 partial pressure of 21 vol % (air), and a roasting time of 80 min.

1. INTRODUCTION

Antimony and its compounds are widely used in making catalysts, ceramics, glasses, batteries, paints, firework materials, ammunition, and fire retardants. Sb consumption is continuously increasing, whereas natural reserves of Sb are declining; thus, the recovery of Sb has become a topic of interest. In nonferrous metallurgy processes, Sb is mainly concentrated in dusts and slags together with As due to their similar chemical properties. This dust cannot be directly used in the Sb smelting process owing to its high As content. In this work, a new method for removing arsenic by roasting As–Sb dust under an oxidizing atmosphere using CaO as an additive is put forward. The influence of the different volatilities of As and Sb compounds, some roasting processes have been developed. Li et al. and Tang et al. found that only approximately 60 mass % of arsenic was removed and more than 10 mass % of antimony was lost when the dust was roasted under an oxidizing atmosphere, possibly due to the formation of an As–Sb solid solution during the roasting process. The vapor pressure of this As–Sb solid solution is lower that of As2O3 but higher than that of Sb2O3, making it difficult to achieve a high As–Sb separation efficiency. Thus, several recent methods for the treatment of As–Sb dust using CuO and MnO2 as weak oxidants have been proposed. In these methods, the structure of the As–Sb solid solution is effectively destroyed, and the arsenic removal rate can reach over 90 mass % with an antimony loss rate of less than 9%. Most Sb is retained in the roasted residue in the form of Sb2O4, and the Cu and Mn are retained in the forms of Cu2O and Mn3O4 respectively. However, this leads to a new problem for recovering Sb from these roasted residues through a general reducing smelting process, as the Cu and Mn will enter the Sb melts, decreasing the quality of the Sb. Thus, for Sb recovery, metal oxides with oxidizing properties should not be used.

In this work, a new method for removing arsenic by roasting the As–Sb dust under an oxidizing atmosphere using CaO as an additive is put forward. The influences of processing parameters, namely, the amount of CaO added, the roasting temperature, oxygen partial pressure, and roasting time, on the separation of arsenic were investigated.

2. RESULTS AND DISCUSSION

2.1. Thermodynamic Analysis. The contents of arsenic and antimony in the dust are high (Table 1) and mainly exist as As2O3, Sb2O3, and As–Sb solid solution ((As, Sb)2O3) in...
Table 1. Chemical Composition of the As–Sb Dust

| Component | As | Sb | O<sup>+</sup> | C | Se | Pb | F | Fe | Bi | Others |
|-----------|----|----|------------|---|----|----|---|----|----|--------|
| wt (%)    | 36.28 | 28.72 | 22.35 | 2.52 | 2.46 | 2.06 | 2.05 | 1.13 | 0.68 | 1.75 |

“Data obtained from a LECO TC-600 nitrogen/oxygen analyzer.

the dust. As shown in Figure 1, the vapor pressure of the As–Sb solid solution is lower than that of As2O3 but higher than that of Sb2O3. Meanwhile, the As2O3 could react with Sb2O3 to form a vitreous material (As–Sb–O) at temperatures higher than 300 °C. For these reasons, it is difficult to separate As2O3 from Sb2O3 effectively via a roasting process under an inert atmosphere. As2O3 and Sb2O3 could be oxidized to nonvolatile compounds as shown in eqs 1–3; additionally, the Sb2O3 oxidation is favored due to the more negative Gibbs free energy values of eqs 1 and 2 compared with that of eq 3. Thus, As2O3 can be effectively separated from Sb2O3 via a selective oxidation process, in which Sb is oxidized and transformed into nonvolatile Sb2O4 and Sb2O5 remaining in the residue, whereas As is volatilized in the form of As2O3(g).

According to eqs 1 and 2, the formation of a vitreous material (As–Sb–O) would inhibit the separation of As2O3 from Sb2O3. The diagram in Figure 3 shows the predominance of As2O3–Sb2O3–O2 as a function of temperature at different As2O3(g) partial pressures, as calculated using the phase stability diagram module of HSC Chemistry 7.0 on the basis of the minimum Gibbs free energy. As shown in the diagram, the Sb(s), Sb(l), Sb2O3, Sb2O5, and 1.5As2O3:0.5Sb2O3 phases are obtained at different oxygen partial pressures and temperatures. It is notable that the stable region of 1.5As2O3:0.5Sb2O3 decreases with the As2O3(g) partial pressure and temperature. Thus, we can infer that the separation of As2O3 from Sb2O3 will also be accelerated at higher temperatures.

2.2. Effects of the Amount of CaO Added. Conditions of air atmosphere (21 vol % O2), a flow rate of 200 mL/min, a roasting temperature of 550 °C, and a roasting time of 90 min were chosen for studying the effects of the amount (5, 8, 12, 14, and 20 mass %) of CaO on the volatilization of As and Sb. In addition, a control experiment without CaO was also carried out under the same conditions.

According to eqs 1 and 2, and Figures 3 and 4a, when no CaO is added, some Sb2O3 could be oxidized to Sb2O4 and Sb2O5 under an air atmosphere, whereas the rest will combine with As2O3 to form the As–Sb–O phase (Figure 5). The “Sb” in Sb2O3 contains Sb<sup>3+</sup> and Sb<sup>5+</sup>. Figure 5 shows that the residue of the dust roasted without CaO is mainly composed of Sb–O and As–Sb–O phases and does not contain any free As2O3 phase. The formation of the As–Sb–O phase obviously restricts the volatilization of arsenic, but promotes the volatilization of antimony as deduced from Figure 1, as a result of which the V<sub>s</sub> is only 70.47%, whereas the V<sub>a</sub> is up to 35.60% as shown in Figure 6.

Based on the data shown in Figures 2 and 4, when CaO was added, the oxidation of free Sb2O3 and the “Sb2O3” component in (As, Sb)2O3 in the untreated dust is promoted, which also inhibits the formation of the As–Sb–O phase. Most of the Sb-containing phases were transformed to nonvolatile Sb2O3, Sb2O5, Sb2O4, Sb2O3, and Ca3Sb2O7 after roasting in the presence of CaO (Figure 7), and massive volatilization of arsenic in the form of As2O3(g) was observed. Correspondingly, the arsenic and antimony volatilization rates increased to 92.69% and decreased to 6.77% (Figure 6), respectively, when 5 mass % CaO was added. However, as the amount of CaO increased further, more Ca3(AsO4)2 was formed through eq 7 (Figure 8), causing a decreases in the V<sub>s</sub> (Figure 6). Moreover, eq 7 is an exothermic reaction (Figure 9) and will increase the temperature in the reaction region, which increases the volatilization of antimony to some extent (Figure 6). Thus, to optimize the separation of arsenic and antimony, the optimal amount of CaO is 5 mass %.

\[
\text{(As, Sb)}_2\text{O}_3 + 2\text{CaO} + 2.5\text{O}_2 (g) \rightarrow \text{Ca}_3\text{Sb}_2\text{O}_7 + 0.5\text{As}_2\text{O}_3(g) \tag{6}
\]

\[
\text{As}_2\text{O}_3 + 3\text{CaO} + \text{O}_2 (g) = \text{Ca}_3(\text{AsO}_4)_2 \tag{7}
\]
2.3. Effects of the Roasting Temperature. Using 5 mass % CaO, effects of the roasting temperature ranging from 400 to 600 °C on the volatilization rates of As and Sb were investigated at an air flow rate of 200 mL/min and roasting time of 90 min.

As the roasting temperature increases, the vapor pressures of As2O3 and Sb2O3 increase and the Va andVs values gradually increase (Figure 10). Figure 11 shows the structures of the residues roasted at temperatures of 400 and 450 °C, both of which are noncrystalline based on the apparent broadening of the XRD peak and are mainly composed of the As−Sb−O phase. The generation of this As−Sb−O phase obviously inhibits the volatilization of arsenic, and the Va values are rather low at 17.12 and 20.06% for roasting temperatures of 400 and 450 °C, respectively (Figure 10). In addition, the X-ray diffraction analysis results of the roasted residues conducted with a step of 1° at the rate of one step per minute (the subgraphs in Figure 11) show that some Sb phases in the untreated dust have been oxidized and transformed to Sb2O4, Sb2O5, and Sb6O13 at 400 °C and 450 °C, causing the Vs values to be as low as 4.33 and 6.08%, respectively (Figure 10). In addition, the X-ray diffraction analysis results of the roasted residues conducted with a step of 1° at the rate of one step per minute (the subgraphs in Figure 11) show that some Sb phases in the untreated dust have been oxidized and transformed to Sb2O4, Sb2O5, and Sb6O13 at 400 °C and 450 °C, causing the Vs values to be as low as 4.33 and 6.08%, respectively (Figure 10). When the roasting temperature was increased above 500 °C, the noncrystalline structure of the roasted residue was destroyed, and the generation of the As−Sb−O phase was inhibited due to the transformation of Sb2O3 and (As, Sb)2O3 in the untreated dust to Sb2O4, Sb2O6, SbO13, and Ca2Sb2O7 (Figure 12). The As volatilization rate (Vv) increases significantly from 35.25 to 91.25% when the temperature was raised from 500 to 550 °C, whereas the Vs changes only slightly from 7.15 to 8.95%. Moreover, the As might be oxidized to As5+ compounds at 600 °C, and the Vs value decreases slightly (Figure 10). Thus, the roasting temperature should be fixed at 550 °C to increase the As volatilization rate.

2.4. Effects of the Oxygen Partial Pressure. The oxygen partial pressure plays an important role in the phase transformation of arsenic and antimony during the roasting process, as shown in Figure 13. In regions of II, III, and V, arsenic exists stably in the form of As2O3, whereas antimony is present as nonvolatile Ca2Sb2O7, Sb2O4, and Sb2O5, allowing for the volatilization and separation of arsenic. When the oxygen partial pressure is increased to the regions of IV, VI, and VII, arsenic and antimony both exist as nonvolatile phases (As2O5, Sb2O4, and Sb2O5), as a result of which arsenic separation is very difficult to be carried out.

Under a N2 atmosphere, most arsenic and antimony in the untreated dust remained in the forms of As2O3 and Sb2O3 (region I in Figure 13) and would be combined into a vitreous material (As−Sb−O) during the roasting process,24 resulting in a low As/Sb separation efficiency (Figure 14). At an oxygen partial pressure of 15 vol %, most phases of (As, Sb)2O3 and Sb2O3 in the untreated dust were oxidized and transformed to Sb2O5, Sb2O6, and Ca2Sb2O7 (Figure 15), through which the formation of the vitreous material (As−Sb−O) was hindered. As a result, the volatilization rates of As and Sb...
increase and decrease to 91.54 and 9.14%, respectively (Figure 14). When the oxygen partial pressure was increased above 21 vol %, some arsenic phases might be oxidized to As$^{5+}$ compounds and the $V_a$ decreases, as shown in Figure 14. To improve the separation efficiency of As and Sb, the most suitable $O_2$ partial pressure is 21 vol % (air).

2.5. Effects of the Roasting Time. As shown in Figure 16, the $V_a$ reaches 91.53% at a roasting time of 80 min and then increases slightly with a longer roasting time, and the Sb volatilization rate remains almost constant at roasting times less than 80 min and then increases obviously at roasting times longer than 90 min. To achieve the aim of improving the As removal rate and decreasing the mass loss of Sb, 80 min is the most appropriate roasting time, at which the Sb volatilization rate $(V_s)$ is 4.30%.

Under the optimum conditions of 5 mass % CaO, a roasting temperature of 550 °C, an $O_2$ partial pressure of 21 vol % (air), and a roasting time of 80 min, the volatilization rate of As was 91.53% and that of Sb was only 4.30%. A high As/Sb separation efficiency was realized in this process, and the As-containing volatiles could be collected and used for preparing FeAsO$_4$, which renders the As harmless. Meanwhile, the Sb in the roasted residue, which existed as Sb$_2$O$_3$, Sb$_2$O$_5$, Sb$_6$O$_1$$_3$, and Ca$_3$Sb$_2$O$_9$, could be reclaimed through a general smelting process.
3. CONCLUSIONS

The effective removal of As from the As–Sb dust by roasting with CaO under an oxidizing atmosphere was feasible. The
amount of CaO, the roasting temperature, and the oxygen partial pressure played significant roles in the As separation.

The As could be volatilized and separated in the form of $\text{As}_2\text{O}_3(g)$ from the As$-$Sb dust through a selective oxidation process from a thermodynamic point of view, and, meanwhile, the Sb was oxidized and transformed into nonvolatile forms (Sb$_2$O$_3$, Sb$_2$O$_5$) remaining in the residue. Furthermore, the oxidation of the “Sb$_2$O$_3$” component in the (As, Sb)$_2$O$_3$ solid solution in the raw dust has traditionally been hindered by the affinity between “As$_2$O$_3$” and “Sb$_2$O$_3$” in (As, Sb)$_2$O$_3$. By adding CaO, this hindrance was alleviated.

A vitreous material (As$-$Sb$-$O) could be formed during the roasting process when CaO was not added and an inert atmosphere was used; the formation of this material would obviously restrict arsenic volatilization. By roasting the As$-$Sb dust at a temperature above 500 °C under an oxidizing atmosphere in the presence of CaO, the formation of the vitreous material (As$-$Sb$-$O) could be avoided through the transformation of Sb$_2$O$_3$ and (As, Sb)$_2$O$_3$ in the untreated dust into Sb$_2$O$_4$, Sb$_2$O$_3$, Sb$_6$O$_13$, and Ca$_3$Sb$_2$O$_7$, and the volatilization of arsenic was promoted. By using the optimum conditions of 5 mass % CaO, a roasting temperature of 550 °C, an O$_2$ partial pressure of 21 vol % (air), and a roasting time of 80 min, an As volatilization rate of 91.53% was achieved, whereas that of Sb was only 4.30%.

4. EXPERIMENTAL SECTION

4.1. Materials.

The As$-$Sb dust was collected from a plant located in the Yunnan province of China, which mainly treats tin anode slime through a pyrometallurgical process. The chemical composition of the dust was characterized by chemical analysis, and the results are given in Table 1. Table 1 shows that the arsenic and antimony contents in the dust are 36.28 and 28.72 mass %, respectively; thus, the recovery of Sb is worthwhile. The XRD pattern of the dust is shown in Figure 17; its main mineral constituents are As$_2$O$_3$, Sb$_2$O$_3$, and a solid solution of (As, Sb)$_2$O$_3$. An electron probe microscopic analysis coupled with energy-dispersive X-ray spectroscopy (EDS) was carried out to detect the elemental distribution in different phases of the dust. Figure 18 shows the focal points of analysis in the raw material, and Table 2 gives the composition of these points. The major phases present in the dust are...
determined to include As−O, Sb−O, and coexistent phases of (As−Sb−O), which was consistent with the XRD results in Figure 17.

The additive CaO was obtained as an analytical purity chemical reagent from Tianjin Shentai Chemical Reagent Technology Co. Ltd., China. In addition, nitrogen (99.3% purity), oxygen (99.3% purity), and air were procured from local suppliers.

4.2. Roasting Methods. All of the experiments were conducted in a horizontal resistance furnace; the schematic diagram of the experimental equipment has been reported previously,26 which is shown as Figure 19. The samples were roasted using a tube furnace (GSL-1500X, Hefei Kejing Materials Technology Co. Ltd., China) under different roasting temperatures, amounts of CaO, O₂ partial pressures, and roasting times. The roasting temperature was precisely measured by a Pt−Rh thermocouple and controlled using a KSY Intelligent Temperature Controller (accuracy: ±1 °C). Nine grams of the dust was used in each experiment. The dust was first ground and sieved to yield a grain size below 200 mesh and then mixed thoroughly with a given amount of CaO. The amount of CaO to be added was calculated based on the mass ratio of CaO to the dust; CaO contents of 0−20 mass % were investigated. For the experimental procedure, the horizontal tube furnace was first heated to the desired temperature and then cleaned using a preset N₂ + O₂ gas mixture. The sample boat containing the samples was weighed and placed in the constant-temperature zone of the furnace when the temperature reached the set value, and was then held for the desired roasting time. Meanwhile, the off-gas from the reaction tube was continuously passed through an As₄O₆ condenser, which collected the volatile matter, and then directed to NaOH solutions to remove harmful components. After the desired roasting time, the sample was cooled under a N₂ atmosphere, weighed once the temperature reached room temperature, and prepared for analysis.

4.3. Characterization. The samples were characterized using X-ray diffraction analysis (XRD, Rigaku, TTR-III), electron probe microanalysis techniques (EPMA, JXA82, JEOL), and scanning electron microscopy (HITACHI-S3400N) coupled with energy-dispersive X-ray spectroscopy (EDS) to study the phase transformation in roasting processes. The X-ray diffraction study was carried out with the 2θ varying from 10 to 90° using Cu Kα radiation at a scanning rate of 8°/min. The chemical compositions of the samples were analyzed using chemical analysis. The software HSC Chemistry 7.0 was used to calculate the standard Gibbs free energy changes of the reaction system. Mathematical expressions for the volatilization rates of arsenic (Vₐ) and antimony (Vₛ) were defined as follows

\[
V_a = \left(1 - \frac{M_a \times W_a}{M_i \times W_{io}}\right) \times 100\
\]

and

\[
V_s = \left(1 - \frac{M_s \times W_s}{M_i \times W_{iso}}\right) \times 100\
\]
where $M_t$ and $M_r$ represent the total mass of the original dust and roasted residue, respectively; $W_{oa}$ and $W_{os}$ are mass percentages of As and Sb in the untreated dust, respectively; and $W_a$ and $W_s$ are mass percentages of As and Sb in the roasted residue, respectively.

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**Notes**

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