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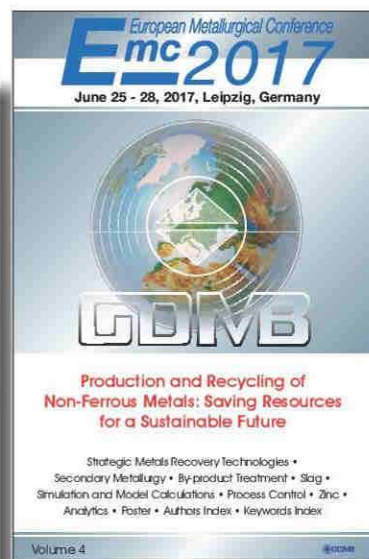
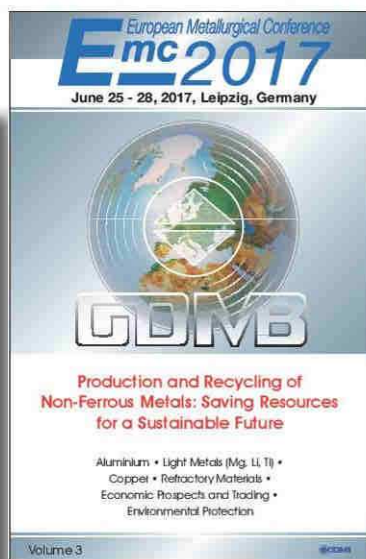
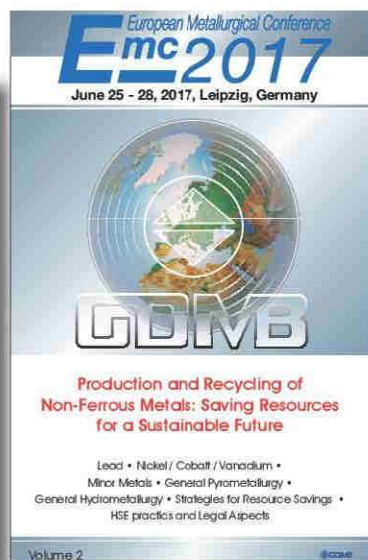
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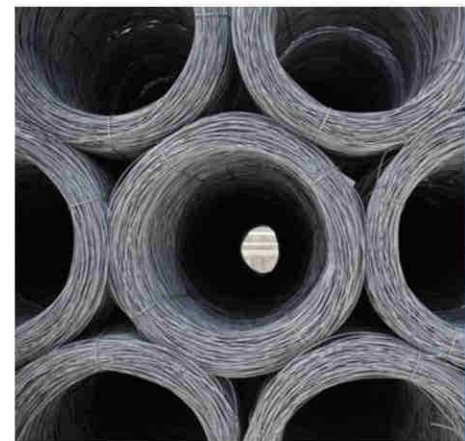
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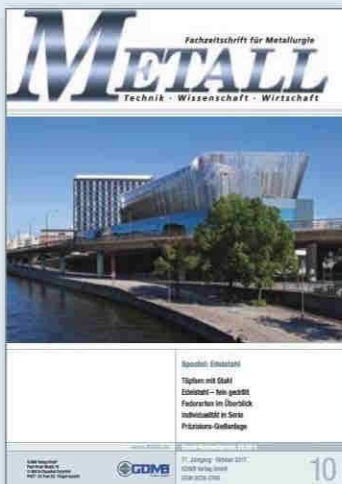
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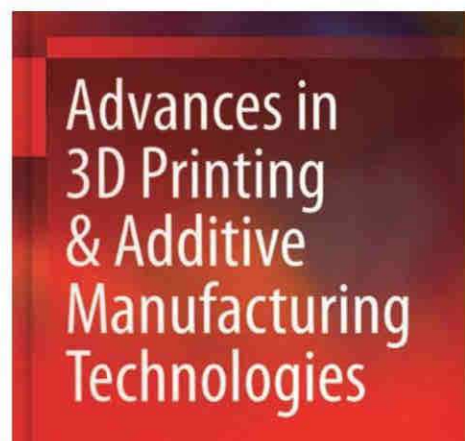
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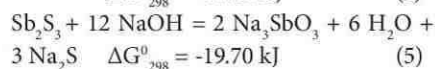
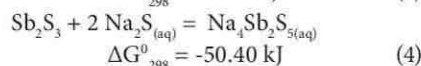
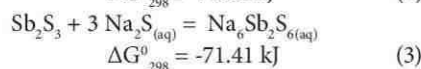
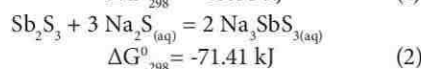
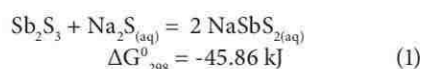
GEOPROFESOURCES

Overview of alkaline leaching of antimony from stibnite

Ružičková, S.; Trpčevská, J.; Sminčáková, E. (1)

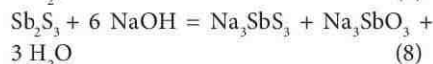
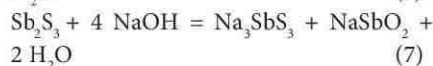
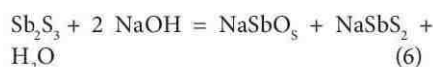
This paper is an overview of alkaline leaching of stibnite and sulphide antimony-containing ores by mixed $\text{Na}_2\text{S}+\text{NaOH}$ or by NaOH leaching solutions. The behaviour of minor constituents, such as mercury, copper, nickel, iron, lead and zinc in alkaline leaching is also discussed in detail. The apparent activation energy and apparent order of stibnite reaction with the solution that contained NaOH were determined from experiments as $E_a \approx 40.5 \text{ kJ mol}^{-1}$ and $n \approx 1$, respectively. Investigations into the rates of leaching stibnite by $\text{NaOH}+\text{Na}_2\text{S}$ solutions, indicated that $E_a \approx 44 \text{ kJ mol}^{-1}$ and the apparent reaction order $n \approx 1.4$, respectively.

The hydrometallurgical route for treating sulphide ores, concentrates, by-products and wastes containing non-ferrous metals is an environmentally friendly method of processing, which is more advantageous than the alternative pyrometallurgical way. One of the important stages in hydrometallurgical processing of natural stibnite (Sb_2S_3), which is often accompanied by other minerals such as silica (SiO_2), pyrite (FeS_2) and wurtzite (ZnS), is leaching, i.e. extraction of antimony into the solution. X-ray diffractograms obtained by X-ray phase analysis of the natural ore were published earlier [1, 2, 3]. Antimony, due to its amphoteric nature, may be extracted from its compounds by either acidic or alkaline solvents [4]. The leaching process can therefore be referred to as alkaline or acidic leaching. During leaching of antimony sulphide, Sb_2S_3 , in Na_2S or NaOH aqueous leaching agents the following reactions take place [5]:



All of the above reactions have negative standard Gibbs energies, which means that all of them are feasible and proceed spon-

taneously. The reaction that has been most frequently referred to in the literature is the reaction (2) in which the antimony sulphide is converted to sodium antimonite when contacted with aqueous sodium sulphide. Leaching by aqueous solution of sodium hydroxide proceeds both according to reaction (5) and according to following reactions [6, 7, 8, 9]:



Leaching process may be represented by predominance area diagrams of $\text{Sb} - \text{S} - \text{H}_2\text{O}$ and $\text{Sb} - \text{S} - \text{Na} - \text{H}_2\text{O}$ systems that were calculated and published by several authors [5, 10, 11]. The diagrams illustrate the complexity of the solution resulting from leaching as it consists of major components, Sb , S , Na , which in aqueous medium form numerous complex ions.

Leaching agent containing Na_2S is a universal solvent for the majority of antimony compounds and, moreover, it has high selectivity for extraction of antimony, i.e. its separation from other accompanying elements. This is not the case of arsenic, tin and mercury [12, 13, 14, 15, 16, 17] as sulphides of these metals react with Na_2S -containing aqueous leaching agent where they form the complex ions such as $[\text{AsS}_3]^{3-}$, $[\text{SnS}_3]^{2-}$, $[\text{HgS}_2]^{2-}$ [18, 19]. Results of laboratory experiments on alkali leaching stibnite carried out and published by [20, 21, 22, 23, 24] confirmed that mercury passes into the solution.

Overview of alkaline leaching

Mixed aqueous leaching medium consisting of $\text{Na}_2\text{S} + \text{NaOH}$ and NaOH aqueous solution are not only applied in leaching of may also be applied in leaching Sb_2S_3 or Sb_2O_3 [5, 7, 25, 26, 27, 28, 29, 30, 31], but have been used successfully in leaching of other minerals present in sulphidic ores or concentrates. The following are examples:

- tetrahedrite – $\text{Cu}_8\text{Sb}_4\text{S}_{12}$ [15, 16, 39, 40, 41, 42, 43, 44, 45, 46],
- enargite – Cu_3AsS_4 [13, 14, 32, 33, 34, 35],
- jamesonite – $\text{Pb}_5\text{Sb}_3\text{S}_{15}$ [14, 32],
- anode sludge with high antimony concentration leaching by KOH aqueous leaching agent [36],
- berthierite – FeSb_2S_4 , boulangerite – $\text{Pb}_5\text{Sb}_3\text{S}_{15}$ and franckeite – $\text{FePb}_3\text{Sn}_3\text{Sb}_2\text{S}_{14}$ [37, 38].

The solution resulting from leaching operation containing antimony may be processed by electrolysis [7, 12, 39], by reduction with hydrogen gas [47], precipitation smelting with iron [48], cementation by aluminum, copper, etc. [8].

The objective of this paper is to assess the leaching process from several aspects based on literature resources with the focus on the following:

- Selection of the most appropriate leaching agent for selective extraction of antimony to aqueous phase.
- Description of behaviour of accompanying elements during leaching, i.e. Cu , Ni , Fe , Zn , Pb and Hg .
- Analysis of antimonite leaching in both mixed ($\text{Na}_2\text{S} + \text{NaOH}$) and NaOH leaching agent from the kinetic point of view and the effect of the following parameters on rate of leaching: solid to liquid ratio, temperature, agitation rate, Sb_2S_3 particle size, concentration of aqueous leaching agent ($\text{Na}_2\text{S} + \text{NaOH}$ and NaOH) and last but not least Na_2S to NaOH ratio in mixed aqueous leaching agent.

Two methods are applicable when studying kinetic of antimony leaching in alkaline medium: rotary disc [49] and agitation leaching.

Experimental

Natural stibnite originating from Pezinok (Slovakia) mine was sampled and subjected to experimental laboratory investigation

of leaching process. The sample chemical composition in weight percents was as follows: 49.35 % Sb, 19.15 % S, 10.4 % Si, 5.43 % Zn, 1.81 % Ca, 0.84 % Fe, 0.65 % Al, 0.63 % Pb, 0.37 % Mg, 0.115 % Cu, 0.015 % Hg, 0.0018 % Ni, 0.038 % As, 0.023 % Sn and traces of Ti, Mn, Bi and Ag. The experimental conditions (temperature, agitation rate, particle size, leaching time, composition of the leaching solution, etc.) are given in respective publications.

Results

X-ray diffraction analysis revealed the presence of the following minerals in the sample: stibnite (Sb_2S_3), quartz (SiO_2), pyrite (FeS_2) and wurtzite (ZnS). Other minerals which would contain minor concentrations of antimony were not detected [1, 2, 3].

Results obtained from kinetic study of leaching by rotary disc method can be summarized in a following way:

- Sb_2S_3 leaching in mixed Na_2S + NaOH leaching agent is more efficient than leaching in leaching agent consisting of only NaOH [50, 51, 52].
- Leaching in mixed Na_2S + NaOH leaching agent resulted in transfer of mercury into solution and the amount of mercury that enters the solution increases with increasing concentration of the leaching agent at constant temperature 297 K [20, 21, 22, 23]. Simultaneous leaching of mercury by mixed alkaline solution was also observed by other authors [15, 18, 53, 54].
- Several parameter have an effect on kinetics of leaching of antimonite:
 - Rate of disc rotation. The optimum rate according to [50, 51, 55] is 3.3 s^{-1}
 - The leaching rate was favourably affected by temperature in the temperature range 297 K to 338 K [50, 51]. The rates of Sb dissolution were plotted in Arrhenius coordinates, $\ln k$ versus $1000/T$ in Fig.1. The resulting apparent activation energy of the process, $E_a \approx 29 \text{ kJ mol}^{-1}$, suggests that antimony leaching is the process controlled by both diffusion and chemical reaction (intermediate process) [56, 57].
 - The leaching rate is enhanced by increasing concentration of Na_2S + NaOH leaching agent in the concentration range from 0.5% Na_2S + 0.5% NaOH to 5% Na_2S + 5% NaOH. The apparent order of reaction decreases

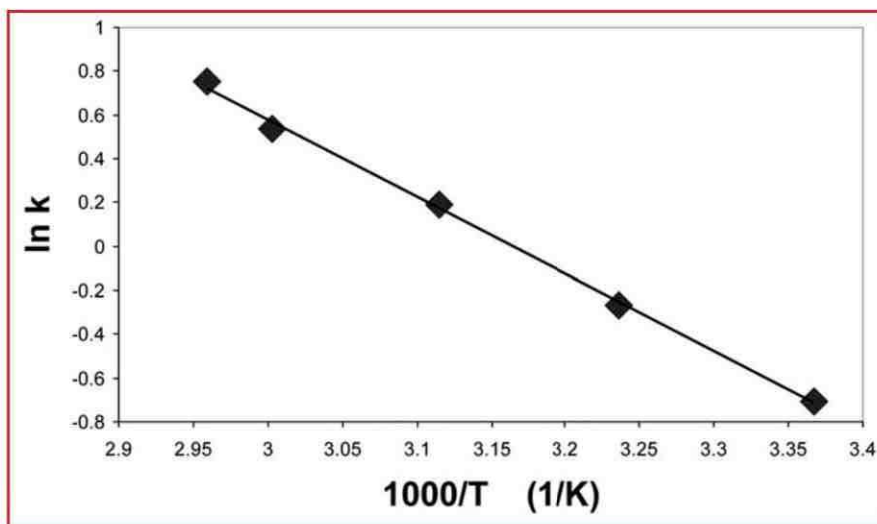


Fig.1: Arrhenius plot for rate of antimony dissolution into the solution containing 1% Na_2S + 1% NaOH at the speed of rotary movement 3.3 s^{-1} , correlation index $r = -0.9984$

with increasing Na_2S concentration in the leaching agent [50, 51, 55].

- By rotary disc method only leaching in 5% NaOH leaching agent was studied. When comparing leaching rates of antimonite by mixed 5% Na_2S + 5% NaOH and NaOH aqueous leaching agents it is obvious that Na_2S has very favourable effect on leaching rate [50, 51].

Results obtained from kinetic study of leaching by agitation method may be summarized as follows:

- Application of mixed 1% Na_2S + 1% NaOH leaching agent resulted in higher antimony recovery (about 88 %) in comparison with either 1% NaOH + 1% $\text{Na}_2\text{S}_2\text{O}_3$ or 1% NaOH (about 42 %) after 30 minutes of leaching at $T = 297 \text{ K}$ [58].
- Alkaline leaching leaves behind nickel and copper in insoluble sludge. The concentrations of other elements in the solution decreased in the order Fe, Pb and Zn; and all of them had less than 1 % recovery.
- Several parameters have an effect on kinetics of agitation leaching of antimonite:
 - Solid to liquid ratio. Optimum ratio was found to be $s : l \leq 1 : 50$ [20, 21, 23].
 - Rate of agitation. Optimum rate $\omega \geq 8.3 \text{ s}^{-1}$.
 - Leaching rate is inversely proportional to the initial antimonite particle size [2, 59].
 - The rate of leaching in mixed Na_2S + NaOH leaching agent is favourably affected by temperature.

The apparent activation energy is $E_a \approx 44 \text{ kJ mol}^{-1}$ [25].

- The apparent order of reaction is $n \approx 1.4$
- Leaching agent concentration. Optimum molar ratio of Na_2S : NaOH in mixed leaching agent is 0.5 [3, 25].
- Investigation of kinetics of leaching in NaOH solution showed that:
 - The lower the initial particle size of antimonite the shorter is the leaching time [9].
 - Between 298 K and 348 K the activation energy $E_a \approx 40.5 \text{ kJ mol}^{-1}$, suggesting that the process is controlled by surface reaction.
 - The apparent order of reaction (6) with regard to NaOH concentration in the concentration range $0.125 \text{ mol dm}^{-3}$ to 1 mol dm^{-3} is $n \approx 1$.

Leaching of antimonite in NaOH and mixed Na_2S + NaOH leaching agent is described by shrinking core model equation [27, 60]:

$$[1 - (1 - X_{\text{sb}})^{1/3}] = k \cdot t \quad (9)$$

where: X_{sb} – antimony conversion [-]
 t – leaching time [min]
 k – rate constant [min^{-1}]

Conclusion

Leaching agent consisting of sodium sulphide and sodium hydroxide can selectively extract antimony from stibnite leaving behind copper, nickel, zinc and lead in the residue. Other minerals of metals such as iron and mercury, which often accompany antimony-bearing mineral resources, may

be partially extracted by leaching, which happens mainly when leaching in Na_2S + NaOH leaching agent with higher concentration. Higher temperature does not remarkably enhance extraction of iron and mercury by leaching. Sodium disulphide aqueous leaching agent fulfills the majority of the requirements for leaching medium: fast leaching rate, high selectivity of antimony extraction and separation from accompanying elements other than arsenic, tin and mercury and last but not least possible recycling of spent electrolyte after electrolytic precipitation of antimony from the solution.

Acknowledgment

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