

An exploration into the sulphide precipitation method and its effect on metal sulphide removal

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Abstract

In hydrometallurgical processes, the use of metal sulphide instead of hydroxide precipitation has gained prominence in recent decades. The arguments for its preferential use are based on the high degree of metal removal at relatively low pH values; the sparingly soluble nature of sulphide precipitates; favourable dewatering characteristics and the stability of the metal sulphides formed. However, when choosing a metal sulphide precipitation route, various difficulties are encountered, two of which are described in the current work.

The first issue is that these metal sulphide precipitation processes, dominated by low solubilities and high supersaturation levels, favour the formation of fine particles due to the dominant mechanisms of homogeneous nucleation, aggregation and attrition.

The second issue is that, in areas of high local sulphide concentration, the excess sulphide can lead to the formation of aqueous polysulphide complexes, which consume the sulphide reagent and compromise the metal removal.

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1. Introduction

In hydrometallurgical processes, the use of metal sulphide instead of hydroxide precipitation has gained prominence in recent decades (Bhattacharyya et al., 1979; Bhattacharyya et al., 1981; Kim, 1981; Veeken et al., 2003). The arguments for its preferential use are based on the high degree of metal removal at relatively low pH values; the sparingly soluble nature of sulphide precipitates; favourable dewatering characteristics and the stability of the metal sulphides formed. However, when choosing a metal sulphide precipitation route, various complexities are encountered, two of which are described in the current work.

The first issue is the nature of the particles formed in metal sulphide precipitation. Because of the extremely low solubilities of most metal sulphide salts, the supersaturation created by the interaction of the aqueous sulphide and metal species can be very high (up to 10^{35} and higher). Thus, the dominant precipitation mechanisms are homogeneous nucleation and limited crystal growth. Attrition of the sulphide crystals due to turbulence, combined with possible aggregation of the solid particles contributes to the formation of fine particles. These can cause problems in downstream processing and should be avoided. This paper presents some preliminary work using both an aqueous and a gaseous sulphide source and compares the results in terms of both conversion and removal efficiency.

The second issue is the formation of aqueous polysulphide complexes under conditions of excess

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sulphide, which can be experienced either globally or locally. This phenomenon was observed in this work when using sulphide to separate copper as a copper sulphide precipitate. The same phenomenon was observed in mixed nickel and cobalt sulphide precipitation under batch conditions. The unexpected result in both cases was that it was not possible to precipitate the metal to the very low levels expected by purely solubility considerations, due to the solubilization of metal in aqueous polysulphide complexes consuming any excess of sulphide.

2. Materials and methods

All experiments were conducted with analytical grade $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ or $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ Merck chemicals. Solutions were made up to required concentrations using distilled water.

2.1. Fluidised bed reactor

The fluidised bed reactor, as illustrated in Fig. 1, consisted of a borosilicate glass column 1.5 m high and 25 mm ID, sealed from the atmosphere. The column was filled to a resting height of 90 cm with beach sand (SiO_2) (250–500 μm) as a seeding material. See [Guillard and Lewis \(2001\)](#) and [van Hille et al. \(2005\)](#) for more details. The aqueous, metal-rich stream together with the recirculation stream entered via the bottom of the column. Sodium sulphide was introduced via three inlet points on the side of the reactor, equally spaced 10 cm from each other, with the first point being 10 cm from

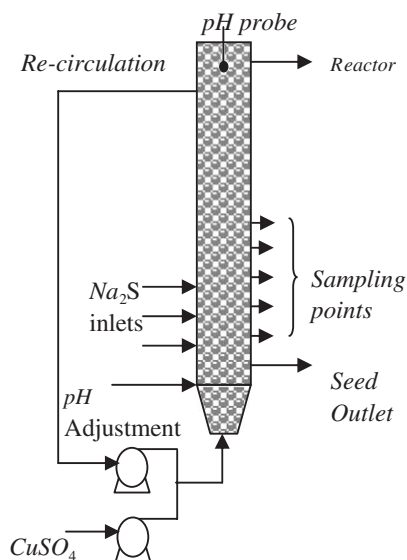


Fig. 1. Schematic representation of the fluidised bed reactor.

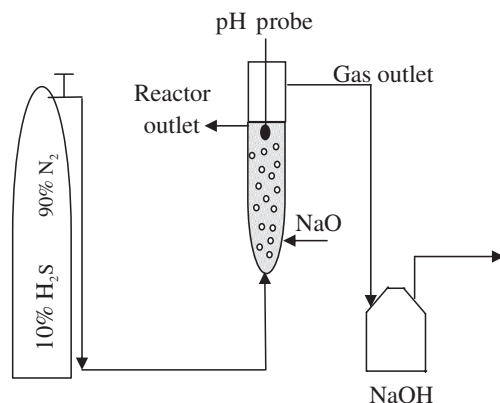


Fig. 2. Schematic representation of the bubble column reactor.

the base of the reactor. The splitting of the sulphide inflow into three was intended to reduce the local supersaturation at the inlet point. Five equally spaced sampling points on the side of the reactor were used to obtain data for column profiles. The first point was situated 15 cm from the base of the reactor with additional points at 10 cm intervals. A sample port 5 cm from the base of the column allowed the removal of coated sand particles for further analysis.

A pH probe (Hanna) was maintained at the level of the effluent port, above the maximum bed height, to monitor the effluent pH. The probe was connected to a custom-built pH controller unit, which helped maintain the pH by controlling the rate of acid (0.1 M H_2SO_4) addition through an inlet port situated 2 cm from the base of the column.

Liquid samples (10 ml), obtained from the sample ports, the recycle stream and the effluent stream, were filtered through a 0.45 μm nylon membrane filter and analysed to determine pH (Cyberscan 2500 pH meter) and metal in solution (Varian SpectrAA-30 atomic

Table 1
Summary of all experiments

Experiment label	Experiment name	Reactor	Cations	Anion
FBR Ni&Co	FBR high and lower supersaturations	Seeded, fluidised bed reactor	Ni and Co	Na_2S
FBR Cu	FBR highest and very high supersaturations	Seeded, fluidised bed reactor	Cu	Na_2S
BC Ni&Co	Bubble column	Unseeded bubble column	Ni and Co	H_2S
B Ni&Co	Batch high and lower supersaturations	Batch reactor	Ni and Co	Na_2S

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