

Recovery of dissolved platinum group metals from copper sulphate leach solutions by precipitation



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ARTICLE INFO

Article history:

Received 29 May 2015

Revised 30 June 2015

Accepted 2 July 2015

Available online 9 July 2015

Keywords:

Extractive metallurgy

Hydrometallurgy

Redox reactions

ABSTRACT

The pregnant leach solution produced in the final leaching stage of base metal refineries (BMRs) operated by platinum producers contains impurities such as selenium and tellurium as well as other precious metals (OPMs, which include Rh, Ru and Ir). The aim of this project was to propose operating conditions for a thio-urea precipitation process that would allow maximum OPM recovery and impurity precipitation from the leach solution with minimal copper and nickel co-precipitation. Experimental results illustrating the effects that operating temperature (80 °C and 160 °C), pressure (atmospheric pressure and seven bar), stirring rate (250 rpm and 500 rpm) and thio-urea quantity (200% and 320% excess) have on the precipitation behaviour are presented.

Virtually all of the Rh contained in the solution was precipitated irrespective of the values of the process variables studied. The maximum percentage Ru and Ir precipitation achieved were 87% and 60%, respectively. Complete Se precipitation was observed at all process conditions, while Te precipitation increased as the operating temperature was increased. Increasing the reagent quantity and temperature did, however, also result in increased copper and nickel co-precipitation.

Regression models were used to perform numerical analyses to determine suitable operating conditions. Predictions with this numerical approach suggested that precipitation with 200% excess thio-urea at a temperature of 80 °C and a pressure of 7 bar would yield 98% Rh, 75% Ru, and 48% Ir precipitation with less than 5% Cu and Ni co-precipitation; these results could be experimentally validated.

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1. Introduction

The base metal refineries (BMRs) operated by platinum producers utilise multiple leaching stages to recover nickel and copper by-products while producing a high grade platinum group metal (PGM) concentrate from nickel–copper converter matte. The first leaching stage achieves nickel dissolution and copper cementation, while the second leaching stage is aimed at complete nickel and copper dissolution. The pregnant leach solution produced in the final leaching stage contains impurities such as selenium and tellurium as well as other precious metals (OPMs, which include Rh, Ru and Ir). Selenium and tellurium are typically removed by precipitation with sulphurous acid prior to electrowinning of copper. While a small percentage of the dissolved OPMs precipitate with the selenium and tellurium, the largest portion remains in solution and is recycled to the first stage leach after copper electrowinning.

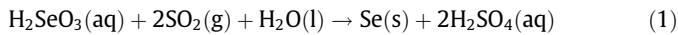
The recycling of OPMs could negatively affect the performance of the process in two ways. Firstly, it causes a relatively large amount of OPMs to be locked up in the process and hence increases the time required to recover OPMs from the converter matte. Secondly, a high concentration of OPMs in the recycled solution places more stringent requirements on the operation of the first leaching stage as any OPMs that are not precipitated will be lost to the nickel product stream. Reducing the OPM concentration in the recycled solution could hence contribute to reduced OPM losses and a reduced OPM plant inventory. Lottering et al. (2012) identified thio-urea as the most promising precipitation reagent for OPM recovery from five reagents investigated (these reagents included sulphurous acid, thio-urea, formaldehyde, formic acid and thiosulphate). The objective of this work was to investigate the effect of key operating variables on the kinetics and extent of OPM and impurity precipitation as well as nickel and copper co-precipitation from a pregnant copper sulphate leach solution when using thio-urea as precipitation reagent. The work also aims to propose suitable operating conditions that could be used to achieve maximum OPM recovery with minimal copper and nickel co-precipitation.

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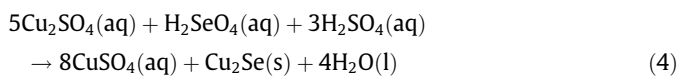
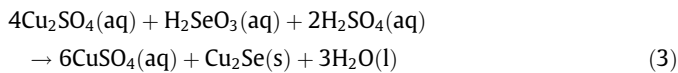
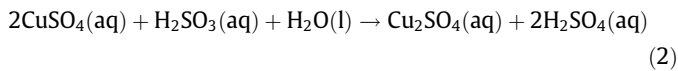
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1.1. Precipitation reactions

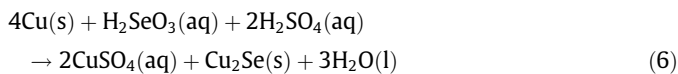
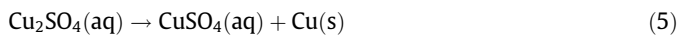
The most commonly used methods for the removal of selenium and tellurium from sulphate leach solutions are precipitation by the addition of sulphurous acid, by the addition of metallic copper, or by addition of a combination of these reagents (Bello and Dorfling, 2014; Hofirek, 1983; Jennings et al., 1969; Shibasaki et al., 1992; Sugawara et al., 1992; Wang et al., 2003; Weir et al., 1982). In the case of selenium, sulphurous acid can reduce tetravalent selenium to elemental selenium according to reaction (1) (Wang et al., 2003):



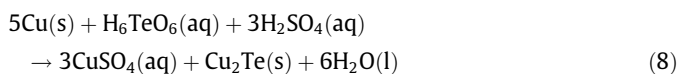
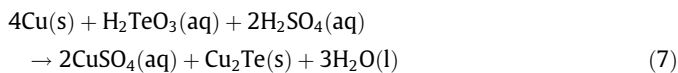
Sulphurous acid also reduces cupric sulphate to cuprous sulphate (Eq. (2)), which subsequently react with tetravalent and hexavalent selenium (Eqs. (3) and (4), respectively) to yield copper selenide (Crundwell et al., 2011).



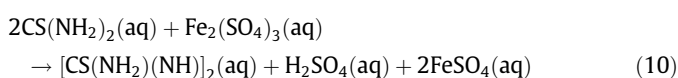
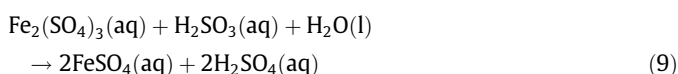
Metallic copper can be added to the leach solution, or it can be formed by the disproportionation of cuprous shown in Eq. (5). Precipitation of selenium as copper selenide by reaction with metallic copper occurs via reaction (6) (Mokmeli et al., 2012; Wang et al., 2003).



Tellurium precipitation generally does not proceed via direct reduction with sulphurous acid or cuprous sulphate, but rather by cementation as copper telluride according to reactions (7) and (8) (Wang et al., 2003):



To the best of the authors' knowledge, reaction pathways for the precipitation of selenium and tellurium from acidic aqueous solutions by thio-urea addition have not been published in literature. It is, however, expected that the reactions occurring when using thio-urea as reducing agent will be similar to the reactions when sulphurous acid is used as precipitation reagent. Published information regarding the behaviour of dissolved iron upon the addition of the different reducing agents supports this notion. Iron present as ferric in the leach solution will consume precipitation reagent according to reactions (9) and (10) for sulphurous acid and thio-urea, respectively (Gupta, 1963):



Thio-urea is also known to react with Cu^{2+} in copper sulphate solutions resulting in the formation of Cu^+ , formamidine disulphide and several other cuprous thio-urea complexes (Fabricius et al., 1994; Szymaszek et al., 1977).

Very limited information regarding the precipitation mechanism of OPMs with sulphurous acid or thio-urea is available in the published literature. Apart from McGeorge et al. (2009), who investigated the precipitation of rhodium from a copper sulphate leach solution by the addition of sodium thiosulphate as precipitation reagent, no further relevant information regarding OPM precipitation behaviour from sulphate leach solutions could be found.

1.2. Effect of operating conditions

Precipitation is dependent on a number of physical and chemical conditions of the process. In order to properly design and optimise control of precipitation process, knowledge on the influence of operational parameters on precipitation process is important (Söhnel and Garside, 1992).

The operating temperature can influence the rates and yields of precipitation reactions. Increasing temperature would increase the equilibrium conversion of an endothermic reaction and decrease that of an exothermic reaction (Fogler, 2006). In addition, the solubility of reactant molecules and the solubility of the products (precipitates) are influenced by temperature. Generally, chemically controlled precipitation reactions are known to be heavily dependent on temperature (Söhnel and Garside, 1992).

Several researchers have investigated the influence of temperature on the precipitation process. Weir et al. (1983) studied the effect of temperature on the rate of Se^{6+} precipitation with metallic copper. For the nickel-copper sulphate solution containing 20–50 g/L sulphuric acid, it was observed that the concentration of Se^{6+} was reduced from 100 mg/L to less than 1 mg/L in less than 30 min when the process was operated at 175 °C whereas at 150 °C the selenium concentration was reduced to the same level in about 60 min. Jennings et al. (1969) and Shibasaki et al. (1992) reported similar observations for tellurium precipitation with metallic copper. Lottering et al. (2012) reported rapid precipitation of Se when using sulphurous acid or thio-urea as precipitation reagent at temperatures of 80 °C, 115 °C and 150 °C. Slightly lower extent of Se precipitation at the highest temperature was attributed to an increase in selenium solubility with increasing temperature, as reported by Weir et al. (1980). The rate and extent of tellurium precipitation were, however, highly temperature dependent.

Lottering et al. (2012) also reported the influence of operating temperature on the precipitation of Rh and Ru using sulphurous acid. The report showed that Rh and Ru precipitation was strongly dependent on the variation of temperature. In their investigation, the average Rh precipitation achieved using sulphurous acid at 80 °C, 115 °C, and 150 °C were approximately 10%, 25% and 40%, respectively.

According to Pohorecki and Baldyga (1983), the precipitation process involves three successive stages. These stages consist of mixing reactants on molecular scale which may result in homogeneous reaction. Homogeneous reactions then produce supersaturation which results in precipitation through the mechanisms of nucleation and crystal growth. It is believed that being the first stage of a precipitation process, mixing can significantly affect the course of the process. Studies that have been done on the effects of agitation on metal precipitation processes show that agitation is an important parameter. Serdar (2011) investigated the effect of agitation on Rh recovery from rhodium-containing waste water by cementation using zinc powder. Without agitating the system, a recovery of 9% Rh was achieved in 10 min; the recovery increased to 39% within the same time range when the solution was agitated.

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