

# Online estimation of impurity ion concentration in solution purification process <sup>★</sup>

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**Abstract:** Online estimation of impurity ion concentration is critical in the optimization and control of solution purification process. Online determination equipments of metallic ions are usually expensive and difficult to maintain in hostile production environments. Oxidation-reduction potential reflects the status of impurity removal reaction and can be monitored online continuously by cheap and easy maintainable oxidation-reduction potential meter. This paper proposed an online estimation approach for impurity ion concentration by taking advantage of the online measurable property of oxidation-reduction potential. The proposed method utilized both domain knowledge, offline day-by-day routine production data and online data. Moving horizon estimation method is used in the estimation. The effectiveness of the proposed approach is illustrated by an experimental study.

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## 1. INTRODUCTION

Hydrometallurgy is a widely used approach for obtaining Special High Grade (SHG, 99.995% pure) metal from their ores in which the valuable metal existed in association with other base metals (Hampel (1968), Habashi (2009)). The conversion from intricately composed ores to pure metal involves a sequence of complex chemical reactions and phases transformations. Typically, a hydrometallurgical process consisted of leaching, solution purification and metal recovery. In the leaching process, by using acid, the valuable metals is liberated from the solid state ores. For the impurity of ores and the unselective nature of leaching, the pregnant leaching solution contains not only valuable metal ions but also ions of the associated metals. The presence of these impurities would cause troubles in metal recovery process, such as large drops of current efficiency during electrowinning, resulting in severe energy waste and downgrade of product quality (Flett (1992), Bøckman and Østvold (2000)). Thus, as a link between leaching

and metal recovery, the function of solution purification process is precisely to reduce the concentrations of these impurities to acceptable ranges.

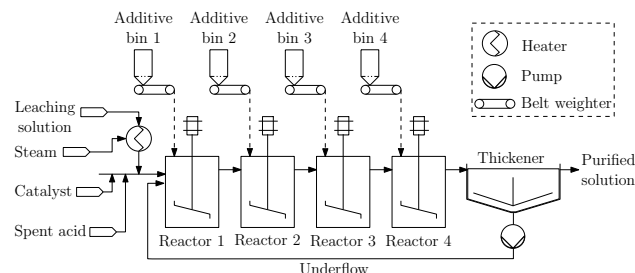


Fig. 1. An impurity removal process

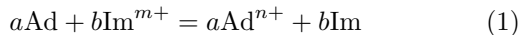
Raw ores contain more than one type of associated metal. As a consequence, solution purification process is composed of multiple impurity removal processes, each of which is designed to remove a particular type of impurity (Sun et al. (2015)). For example, in zinc hydrometallurgy, solution purification process is composed of copper removal (Zhang et al. (2016b)), cobalt removal (Sun et al. (2014)) and cadmium removal. Impurity removal process involves use of additive to replace the impurity ions under specific reaction conditions. As shown in Fig. 1, impurity

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removal is conducted in consecutive continuous stirred reactors. Additive is supplied to remove the target impurity by forming alloys or metal compounds under the assistance of catalyst. Spent acid and steam are also supplied to adjust pH and temperature of the solution. After retention in the consecutive reactors, the solution flows into the thickener in which liquid-solid separation takes place. Overflow of the thickener is filtered and delivered to subsequent process, while the underflow which contains the reaction products, e.g., crystal nucleus, beneficial to impurity removal is recycled to promote purification (Fig. 1).

Influent and effluent impurity ion concentrations of each reactor are key indicators in the determination of additive dosage and configuration of reaction conditions. However, online determination equipments of metallic ions are usually expensive and difficult to maintain in hostile production environments (Wang et al. (2016)). As a compromise, impurity ion concentrations are usually determined periodically (the sampling period is counted by hours) by time consuming artificial chemical assay. So the operators can only get access to the delayed and discrete information of impurity ion concentrations. Consequently, the operators prefer to handle impurity removal process in a conservative manner, i.e., use excessive amount of additive to achieve the required purification performance, which is an increase of production cost.

Building nominal kinetic model offline and updating model parameters online is a rational approach to obtain the realtime information of key performance indicators (Zhang et al. (2013), Xie et al. (2015)). Without loss of generality, in impurity removal process, the reaction of using additive to replace the impurity ions can be expressed as:



in which Ad is the additive, Im is the impurity,  $a, b, m, n \in \mathbb{N}^+$  and  $bm = an$  (Sędzimir (2002)). Reaction (1) is essentially an oxidation-reduction reaction whose extent of reaction is indicated by Oxidation-reduction potential (ORP). ORP represents the comparative oxidability or reducibility of the solution. It can be monitored online continuously using ORP meter which is cheap and easy to maintain. Due to these advantages, it has been applied to built monitoring and control systems of many biological, chemical and hydrometallurgical processes, such as reject water treatment (Yu et al. (2013)), biological nitrogen removal (Ruano et al. (2012)), uranium extraction (Ram et al. (2011)), copper removal (Zhang et al. (2016a)), etc. Sun et al. (2013) studied the relationship between ORP and reaction rate of cobalt removal process, built an integrated prediction model, and applied ORP in the prediction of cobalt ion concentration. The model shows satisfying predict performance. However, the online information of ORP has not been fully utilized in the estimation.

An interesting alternative which circumvents and exploits the use of a model in conjunction with a limit set of measurements is the use of state observers (Dochain (2003)). In the case of linear systems, the standard solutions are the Kalman filter and Luenberger observer (Liu (2013)). As for nonlinear systems, the Kalman filter and the Luenberger observer can be extended based on repeated linearization of the nonlinear system around a reference trajectory.

However, the approximation error caused by the linearization can generate possibly large bias in the estimation. A detailed review of state observers is outside the scope of this article. The readers are suggested to read an excellent review written by Ali et al. (2015). Moving horizon estimation (MHE) has become popular in recent years (Liu (2013)). It has been suggested as a practical tool to handle explicit nonlinear systems and incorporate inequality constraints in estimation. The basic strategy of MHE is to reformulate the estimation problem as quadratic program using a moving, fixed-sized estimation window (Rao et al. (2001), Liu (2013)). So the problem size won't grow with time. The 'old' data outside the estimation window is summarized using arrival cost which is included in the cost function of the batch least square problem. The stability problem of MHE has also been extensively studied (Rao et al. (2003), Voelker et al. (2013), Wynn et al. (2014)).

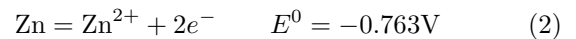
The aim of this paper is to apply MHE in the estimation of impurity ion concentration by using the online information of ORP. Both domain knowledge, offline day-by-day routine production data and online data are used in the estimation. The rest of this paper proceeds as follows. In Section 2, a kinetic model of impurity removal process is constructed by applying the kinetics of electrode reactions. The kinetic model is reformulated to reduce correlations among parameters prior to the identification of kinetic model parameters. In Section 3, the estimation problem of impurity ion concentration is formulated. A nonlinear moving horizon estimation approach is applied in the estimation. In Section 4, the feasibility and performance of using the online information of ORP to estimate impurity ion concentration is investigated through an experimental study. The concluding remarks are given in Section 5.

## 2. OFFLINE STAGE: KINETIC MODEL CONSTRUCTION AND PARAMETER IDENTIFICATION

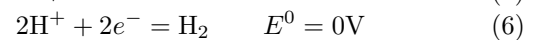
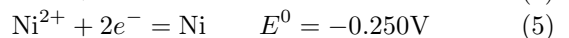
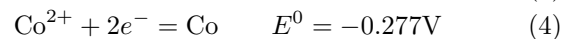
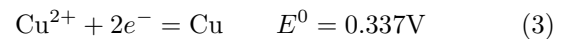
### 2.1 Kinetic model construction

Reaction (1) is an oxidation-reduction reaction as well as an electrode reaction. Due to the complex nature of hydrometallurgical processes and the heterogeneity of ores, there are some parallel electrode reactions taken place simultaneously. Taking cobalt removal process for example (Sun et al. (2013)),

In the anode:



In the cathode:



in which  $E^0$  is the standard equilibrium potential of the electrode reaction. According to the independence principle of parallel electrode reactions (Antropov (1977)) and electrode reaction kinetics (Zha (2002)):

- Electrode reactions are independent of one other, their unique shared characteristic is the electrode potential, which is called the mixed potential  $e_{\text{mix}}$ ;

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