



On-line monitoring of aqueous base metal solutions with transmittance spectrophotometry



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ABSTRACT

Transmittance spectrophotometry was used to monitor copper, cobalt and zinc in solution in laboratory experiments. The samples simulated plant conditions encountered on the Skorpion zinc mine in Namibia and were prepared using a simplex centroid mixture design. Principal component, partial least squares and support vector regression models were calibrated from visible and near infrared absorption spectra. All models could accurately estimate the concentrations of all the metals in solution. Although these models were affected by nickel contamination, the Cu models were less sensitive to this contamination than the Co and Zn models. Likewise, elevated temperatures led to degradation of the calibrated models, particularly the Zn models. The effects of these conditions could be visualized by a linear discriminant score plot of the spectral data.

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1. Introduction

Efficient operation of hydrometallurgical plants often depends on routine analysis of valuable elements after every unit operation (Gaft et al., 2007). In this respect, the availability of on-line data is a critical element in process control and continuous process improvement, as suggested by Sowerby (2002) and Gaft et al. (2007). Since on-line measurements are typically non-intrusive, there is no interference with the process, and thus plant operations can run without interruption. In addition, on-line analysis reduces the health hazards that may be experienced during the collection of samples for laboratory analysis (Sowerby, 2002). Moreover, laboratory analysis could be subject to large sampling errors, whereas on-line methods usually provide high quality sampling data through the analysis of large continuous volumes of material (Gaft et al., 2007).

Several techniques are available for on-line analysis. These include on-stream analysers, image analysis and other optical sensors. In general, on-stream analysers are effective in monitoring and controlling plant processes, since they can determine the elemental composition of a sample with high accuracy (Remes et al., 2007). Optical fibre sensors in particular have shown considerable promise as a means to obtain elemental analyses in the areas of

food processing, pharmaceuticals and mineral processing (Lewis et al., 2007). Even though the method of analysis is not the same in each case, the operating principles underlying the measurements are the same. Different wavelengths typically ranging from ultraviolet (UV) to near infrared (NIR) reflected from the sample are measured by an optical fibre sensor. These reflection patterns can usually be related to the characteristics of the sample material. Recent studies have shown that visible to near infrared (vis-NIR) spectra can be used as a supplementary information for accurate monitoring and control of large mineral plants (Haavisto and Kaartinen, 2009). The benefit is a potential increase in stability in plant operations that can lead to improved plant performance and higher financial returns (Haavisto et al., 2008). Despite its potential, online spectrophotometry is not a mature technology in the metallurgical industries yet, as some of the equipment may be sensitive to external disturbances and would therefore require a high degree of maintenance (Bergh et al., 2001).

The optical sensor used in this study was developed by Blue Cube Systems, based in Stellenbosch, South Africa and is among other used to monitor zinc purification in the Skorpion zinc mine in southwest Namibia. Although the sensor can be used as both a diffuse reflectance and a transmittance spectrophotometer, only the latter mode was considered in this study to simulate implementation on the plant as closely as possible. Zinc operations in the Skorpion complex comprise an open pit mine, mill, an electro-hydrometallurgical refinery, as well as a melting and casting shop. The essential processing steps of zinc production consist of

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roasting of the zinc concentrates from the mine to convert the zinc sulphides into oxides. Sulphur dioxide is converted into sulphuric acid that is used to leach the zinc oxide to produce an impure zinc sulphate solution. The solution is subsequently purified by precipitation of copper with zinc dust in a first stage, to limit the copper concentration in solution to 0–2 mg/L. Other impurities are removed in a subsequent stage and the purified zinc sulphate solution is further processed by electrowinning, where zinc metal is recovered from electroplated aluminium cathodes.

At this site, the spectrometer is applied in the first stage of solution purification, where it is used to measure the concentration of copper in solution in the refinery. This requires periodic recalibration of the sensor, and the quality of the models can have a significant influence on the real time monitoring of the process. Moreover, the prediction of the copper concentration may be influenced by the presence of other metals in solution, such as cobalt and cadmium and in this investigation the effect of some of these metals on the calibration model of the instrument is also investigated.

The data processing and interpretation in this study were done by evaluating and assessing the quality and performance of principal component (PCR), partial least squares (PLSR) and support vector regression (SVR) models to simultaneously quantify copper (Cu), cobalt (Co) and zinc (Zn) in a sample solution. All models were based on the vis-NIR spectra of standard solutions, while the effects of process conditions, such as temperature and contaminants were evaluated and determined under laboratory conditions.

2. Experimental work

2.1. Experimental design

Statistically designed experiments provide a mathematical framework for changing all significant variables or factors simultaneously with a minimum number of samples (Anderson-Cook and Montgomery, 2008). Design Expert v8 software was used to generate a simplex centroid mixture design, as shown in Fig. 1, in which three factors, i.e. the concentrations of copper, cobalt and zinc were varied. The design points (blue circles)¹ correspond to the composition of the calibration samples used in the derivation of the models. In this design, the measured response was assumed to depend only on the relative proportions of the components in the mixture, not the amount or quantity of the mixture (Cornell, 1981).

Permutations of the pure blends are located at the vertices, the binary blends are located along the edges and permutations of the three-component blends are located inside the triangle. The centroid point was repeated twice to yield 12 runs for the design. The values given in the figure are the percentage of the metals from their respective stock solution concentration, as described in Section 2.2. The experimental runs for each set of samples were performed randomly to accommodate any instrumental error that might have occurred during measurement.

2.2. Sample preparation

The sulphate salts of copper, cobalt and zinc metals were used to prepare the stock solutions with concentration values of 500 mg/L, 50 mg/L and 50,000 mg/L, respectively. Each salt was first dissolved in 100 mL of distilled water and then 50 mL of 98% sulphuric acid was added to give a 500 mL solution after further dilution. High purity sulphate salts were used to avoid impurities

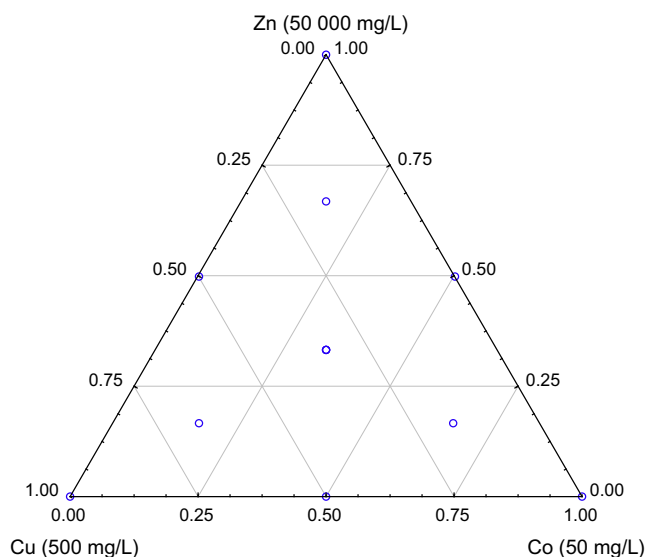


Fig. 1. Three factor simplex-centroid mixture design with data points.

in the metal solutions interfering with the absorption spectra of the metals. The calibration samples were prepared by diluting the stock solutions with 10% sulphuric acid to obtain the required composition of the mixture, as indicated by the design points in Fig. 1.

In order to test the models and evaluate the effect of interference on the predictive ability of the models, the proportions of the metals were chosen arbitrary so that the percentage of total metals added up to 100. In addition, the temperature of the sampling solution was increased from 20 to 80 °C in 20 °C increments to assess the effect of temperature on the performance of the calibration models. To simulate the effect of nickel contamination, nickel was added in increments of 50 mg/L to the sampling solution in concentrations ranging from 50 mg/L to 200 mg/L. The experimental runs are summarized in Tables 1–4.

2.3. On-line measurement set-up

The Blue Cube instrument used for analysis consists of four basic components, i.e. an interface, data processor, sample holder and spectrophotometer. The interface is connected to a main switch and it supplies a steady 12 V to both the data processor and spectrophotometer. The data processor contains the computer that processes the data captured from the sample. The spectrophotometer has an optical sensor with a scratch resistant sapphire window that uses the principle of diffused reflective spectroscopy over a wavelength range from approximately 350–1050 nm (Lottering and Aldrich, 2006).

A light source forms an integral part of the spectrophotometer and light is transmitted from the source to the sample holder via 6 light + 1 dark (reference) 400 µm diameter optical fibres, which are also used to transmit the resulting signal from the solution to the data processor for interpretation. The 100 W instrument is controlled at a temperature of between 50 and 55 °C. Fig. 2 shows a photograph of an industrial prototype of the Blue Cube optical fibre sensor instrument with the features mentioned above. The solution was poured thorough a funnel into the sample holder, with a volumetric capacity of 180 mL. The plastic clamp, shown in Fig. 2, was used to reduce the flow rate of the samples by constraining the inlet tube leading to the sample holder.

The solution inside the measuring cell was allowed to stabilize for 30 s before analysis was done. The start button on a remote

¹ For interpretation of color in Figs. 1 and 10, the reader is referred to the web version of this article.

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