



Determination of the concentration of MIBC in coking coal flotation

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ABSTRACT

This paper presents a novel method to measure the concentration of methyl isobutyl carbinol (MIBC), a widely used frother in coking coal flotation. It is based on the specific interaction between MIBC molecules and a formulated liquid mainly comprising cyclodextrin and pyrene. The method involves mixing the formulated liquid with the sample solution for a short period of time before measuring the absorbance using a UV–Vis spectrophotometer. It shows high measuring accuracy with minimal interferences from diesel, flocculants and inorganic salts present at typical concentrations in coking coal flotation plants.

1. Introduction

Raw coal is often subjected to beneficiation processes to remove excessive impurities before being sold to market. Among the beneficiation methods, froth flotation is widely used for fine size fractions (typically < 1 mm). Froth flotation for coal cleaning has become increasingly more important as more and more fines are generated owing to application of advanced and automated mining techniques. For instance, fines account for around 20 wt% of the raw coal feed in a typical hard coking coal processing plant in Australia (Mackinnon and Swanson, 2010) and up to 40 wt% of raw coal feed on a worldwide basis (Aplan and Arnold, 1991). Despite its importance, the control of froth flotation is poor, largely owing to lack of tools to measure critical process parameters in real time.

One of the most important process parameters for a coal flotation operation is the concentration of frother. Use of frother at appropriate concentration levels can produce desirable bubble size and stability and mobility of the froth phase, which in turn significantly affect the kinetic viability of the flotation processes and its separation efficiency. Excessive residual frother in plant water could cause over frothing (so-called ‘froth out’) problems in pumps, sumps and thickeners. On the other hand, insufficient frother addition in the flotation process often leads to loss of yield (Stevenson and Lambert, 2012). Therefore, regular frother concentration measurement throughout the flotation and plant water circuit is required to identify appropriate dosing rate in order to maintain the flotation performance at a high efficiency (Zangooi et al., 2010; Zangooi, 2014; Zangooi et al., 2017). In other words, it is essential to continuously monitor the frother levels in flotation cells and in plant water circuits to attain maximum efficiency. There is, however,

no fast frother concentration measuring tool to minimize the time delay between actual processes changes and the control actions.

The existing techniques for measuring frother concentration are not effective. Calculating frother concentration based on the dosing amount of frother is difficult in the operating plant owing to incomplete dissolution and unknown distribution of frothers between flotation pulp and froth (Zangooi et al., 2010). In addition, the typical dosages of the frother are too low to make any noticeable changes in surface tension, so it is rather difficult to determine frother concentration from the surface tension measurement. Some researchers used gas chromatography (GC), high-pressure liquid chromatography (HPLC) and NMR spectroscopy with total organic carbon analysis to measure frother concentration (Fuerstenau, 1982; Aston et al., 1989; Tsatouhas et al., 2006; Gredelj et al., 2009; Zanin et al., 2009; Zhang et al., 2013; Noble et al., 2016). These tools are expensive to install and need specialist training to operate and maintain. They also require labour and time-consuming sample preparation prior to the analysis, which causes a relatively long time to complete the analysis. Finch and co-workers developed a colorimetric technique to measure frother concentration (Gélinas and Finch, 2005, 2007; Zangooi, 2014; Zangooi et al., 2010, 2017). It is based on the Komarowsky reaction, which involves the interaction of the frothers, concentrated sulphuric acid, and salicylaldehyde to yield coloured solutions that can be analyzed by UV–visible spectrophotometry. The advantages of this colorimetric technique include high reproducibility, high sensitivity and broad applicability for various frothers. However, low analysis rate and requirement of some degree of human intervention during sample preparation limit its use in online applications.

Research efforts have also been made by others to develop non-

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analytical techniques to measure frother concentration online, ranging from breathalyser-type alcohol detectors (Hart et al., 2006), which are limited to relatively high concentrations, to froth height measurement using a column, which has poor reproducibility (Lahey and Clarkson, 1999). A technique based on gas holdup was also developed to estimate the frother concentration (Weber et al., 2003; Maldonado et al., 2010). However, Maldonado et al. noted that the linear relationship between gas holdup and frother concentration holds only at a limited concentration range (i.e., 5 ppm to 25 ppm MIBC) and the estimated concentration may be incorrect when the concentration of organic and inorganic contaminants varies after calibration. More recently, Kracht and Hunt (2016) proposed a new technique, which utilizes a hydrophone and amplifier to measure acoustic emissions generated during bubble generation and coalescence. Based on a previous study of theirs (Kracht and Rebolledo, 2013), this technique has drawbacks similar to other non-analytical techniques.

In the present study, we demonstrate a novel method involving the mixing of the sample solution and formulated liquid to measure the concentration of MIBC, a short chain alcohol commonly used as frother in coal and metalliferous ore flotation process (O'Connor et al., 1994; Firth, 1999; Pugh, 2007; Firth, 2015). It is based on the specific interaction of MIBC molecules with the formulated liquid mainly comprising β -cyclodextrin (β -CD) and pyrene, which can form a complex with MIBC. The number of the formed complexes is dependent on MIBC concentration, which can be readily quantified by an optical technique (Hamai, 1989). Various studies (Nelson et al., 1988; Hamai, 1989; Munoz De La Pena et al., 1991; Udachin and Ripmeester, 1998) found that ternary complexes would form when alcohols were added to the solution where pyrene and β -CD had already formed a complex. The newly formed ternary complex causes a significant change in the spectra, which can be monitored using a UV–Vis spectrophotometer or a spectrofluorometer. These studies used, however, a high concentration of alcohol aiming at determination of the formation constant and stoichiometry ratio of the complexes. The present work focusses on determination of relatively low concentrations of MIBC in coking coal flotation where other chemical substances, such as diesel, flocculant (a residue from reused plant water), and inorganic electrolytes, might be present.

2. Materials and methods

2.1. Materials

Pyrene (99% pure), α -CD (98% pure), β -CD (97% pure) and methanol (99.9% pure, spectrophotometric grade) were purchased from Sigma Aldrich. A cationic flocculant (HiCat Cl9003) was supplied by Concept Controls, Australia. MIBC (98%, Sigma Aldrich) and diesel (Caltex) were used as frother and collector, respectively. The inorganic salts used for preparing the simulated process water included $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (99%, Chem-Supply), Na_2SO_4 (99%, Chem-Supply), $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (99%, Chem-Supply) and NaCl (99.5%, Sigma Aldrich). All reagents were used as received.

De-ionized water (18.2 M Ω cm) produced from a Milli-Q system (Millipore) was used throughout the experiments.

2.2. Preparation of solutions

In preparing different formulated liquids, two different stock solutions of pyrene were prepared:

- I. Dissolving excessive amount of pyrene into the de-ionized water, which was allowed to equilibrate for more than 48 h at room temperature. Any floating micro-crystals were removed and the remnant solution was used. In determining the concentration of pyrene in the formulated liquid, the water solubility of the pyrene at 27 °C was assumed 0.780 $\mu\text{mol/L}$, the mean of two reported values: 0.815

(Pearlman et al., 1984) and 0.744 $\mu\text{mol/L}$ (Reza et al., 2002).

- II. Dissolving excess amount of pyrene into methanol-water mixtures (i.e., 5, 10, 15, 35, 55 v/v %), which were allowed to equilibrate for more than 24 h. The solubility of the pyrene in these methanol-water mixtures with 5, 10, 15, 35 and 55 v/v % methanol were assumed 1.33, 2.13, 3.42, 22.68 and 150.15 $\mu\text{mol/L}$, respectively (Fan, 1997).

The prepared pyrene stock solution was wrapped with aluminium foil and stored in darkness to minimize the photochemical degradation of pyrene (Clark et al., 2007). The pyrene stock solution showed no significant photochemical degradation over three weeks based on the UV–Vis absorption spectra of the final solutions.

Other stock solutions were prepared by adding a suitable amount of the corresponding chemical into the de-ionized water. These stock solutions included 0.0159 M β -CD and 100 ppm flocculant. Saturated solution of diesel was prepared by adding excessive amount of diesel into the de-ionized water and allowing it to equilibrate for more than 48 h. The remnant aqueous phase was used as a stock solution. In determining the diesel concentration in a sample solution, the water solubility of diesel was assumed 3.2 mg/L (Shiu et al., 1990).

A stock electrolyte solution was made by mixing 0.493 g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 0.142 g of Na_2SO_4 , 0.147 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, and 0.584 g of NaCl with 100 ml of de-ionized water. A 10-fold dilution of this stock electrolyte solution with de-ionized water allowed us to obtain the simulated plant water, whose chemical composition is shown in Table 1. The chemical composition of the simulated plant water is close to that of process water from a coal preparation plant in Australia (Alam et al., 2011).

2.3. Analytical procedures

Fig. 1 conceptually illustrates the analytical method for measuring the MIBC concentration of a sample solution. It involves mixing the sample solution with a formulated liquid for a pre-set period of time, followed by instant UV–Vis absorbance measurement. In most cases, the formulated liquid was a mixture of β -CD and pyrene stock solutions. It was possible to add another chemical into the formulated liquid to suppress the disturbing effect of diesel (see Section 3.3.2).

For the purpose of establishing calibration curves, a 1000-ppm stock MIBC solution was used to prepare the standard sample solutions. Typically, each standard sample solution with known MIBC concentration was mixed with a formulated liquid. In some tests, flocculant, collector, and inorganic salts were introduced into the standard sample solution to mimic the chemical composition of process water in some flotation plants. The final solution (i.e., a mixture of formulated liquid and sample solution) was agitated at 180 rpm using a shaker incubator (TU-453, M.R.C) for 15 min, unless stated otherwise. The absorbance of the final solution was then measured immediately using a UV–Vis spectrometer (Cary 50, Varian) at an ambient room temperature, which was controlled at 23 ± 1 °C by using an air conditioner.

Table 1

Concentration and type of electrolytes used to produce the simulated process water. The chemical composition of process water from a coal processing plant in Australia is shown for comparison (adopted from Alam et al., 2011).

	This study (M)	Alam et al. (M)
Ca^{2+}	1.0×10^{-3}	9.1×10^{-4}
Mg^{2+}	2.0×10^{-3}	1.7×10^{-3}
Na^+	1.2×10^{-2}	2.1×10^{-2}
K^+	–	2.0×10^{-4}
SO_4^{2-}	3.0×10^{-3}	3.7×10^{-3}
Cl^-	1.2×10^{-2}	1.8×10^{-2}

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