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Flow mapping of full scale solvent extraction settlers using pulsed Doppler UVP technique

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HIGHLIGHTS

- A technique for the measurement of fluid flow patterns in opaque solutions presented.
- This believed unique capability is achieved using pulsed Doppler techniques.
- Application in commercial solvent extraction (SX) operations demonstrated.
- Variation in SX settler flow patterns observed with settler 'furniture' modification.

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ABSTRACT

Details behind the experimental technique of ultrasonic velocity profiling (UVP) to obtain *in situ* velocity measurements of both aqueous and organic phases in commercial solvent extraction SX settlers are presented. The commercial applicability and benefit of the process is demonstrated via the on-site analysis of two settlers at a commercial copper solvent extraction operation. The two settlers had the same dimensions and were operated under essentially equivalent conditions but differed in terms of being assessed before and after routine maintenance, with the internal 'furniture' configuration of the latter also being modified. UVP-determined organic and aqueous flow pattern results from the two settlers are reported and compared in relation to the furniture configuration/maintenance status of each. The results highlight the potential for UVP analysis to ascertain flow patterns in commercial solvent extraction settlers to benefit industrial operations directly and to enable the development of improved mathematical (computational fluid dynamics) modelling of flow patterns in settlers.

1. Introduction

1.1. Solvent extraction

Liquid–liquid separation or solvent extraction (SX) is an important step in hydrometallurgical processing of base metals which facilitates selective separation of one or more metal ions of interest from an aqueous solution. It relies on the immiscible nature of organic (typically kerosene-based) and aqueous phases. The organic phase contains an extractant which, under suitable operating conditions, is capable of removing metal ions from the aqueous phase into the organic phase and, under strip conditions, transferring them from the organic phase back into a different aqueous phase. There are various extractants used commercially in SX operations for different metal recovery processes. For instance, the SX extractants of choice for copper operations are phenolic hydroxyoxime-based. These are capable of extracting copper via the reaction shown in the following:

$$Cu^{2+} 2LH_{(org)} \rightleftharpoons CuL_{2(org)} + 2H^{+} \quad (1)$$

where LH represents the neutral hydroxyoxime extractant and the subscript 'org' indicates the species is present in the organic phase. It can be seen from Eq. (1) that this equilibrium reaction will be affected by the operating pH, and this is indeed used to control whether extraction or stripping takes place.

An acceptable rate of metal ion transfer between the immiscible phases in SX is achieved by their vigorous mixing which results in an unstable emulsion containing droplets of one phase in the other, being either aqueous droplets in the organic phase ('organic continuous'), or organic droplets in the aqueous phase ('aqueous continuous'). The smaller the droplet size, the greater the rate of metal ion transfer (Miller, 2006). However, subsequent to mixing, the phases need to be separated as completely as possible to minimise contamination and/or wastage during the subsequent

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purification steps. As per Stokes law, smaller droplets separate more slowly than larger droplets and as such require a longer residence time for separation if increased entrainment is not to result. The conflicting demands of the mixing and subsequent separation stages are an integral and often repeated step in SX operation. The type of equipment and how it is used in these processes is therefore extremely important as it impacts the mixing and separation phenomena and thus the operation of the plant.

1.2. SX contactors and phase separation

There is no single SX contactor that is best for all situations. The metal transfer kinetics along with dispersion and coalescence behaviour can influence the choice of contacting equipment. Continuous contacting equipment used in the industry for solvent extraction can be broadly classified into two groups; stage-wise or differential, based on their mode of operation (Ritcey and Ashbrook, 1979). Mixer–settlers and inline mixers are examples of stage-wise contactors wherein the aqueous and the organic phases are mixed and then separated in a large settling area before the next stage. Columns and centrifuges are classified as differential contactors requiring a smaller area ('footprint') than mixer–settlers but more height.

Mixer–settlers are currently used in a large number of plants as they are relatively easy to operate, reliable, flexible and fairly simple to design. The liquid–liquid mixture is created within the mixer box with the aid of a suitable impeller. In many operating plants, the mixer box is fitted with a pump mixer which, in addition to creating the droplets, also works to draw the organic and aqueous solutions into the mixer box. As requirements for the mixers differ, appropriate impeller design is required to maximise mass transfer as well as decrease entrainment (Kehn and Kontur, 2011). Advances in this area also include DOP® (Dispersion Overflow Pump) pumping unit and SPIROX® mixing unit from Outotec that controls the formation and the size of the generated droplets (Hakkarainen et al., 2011).

In many operating plants, an auxiliary or a secondary mixer which receives the overflow from the primary mixer box also works to provide additional mixing to prevent phase separation or conversely facilitate some coalescence before the mixture overflows into the settler. The unstable and therefore temporary emulsion emanating from the mixer is then with time able to naturally separate out into discrete organic and aqueous layers, with the droplets of the discontinuous phase also coalescing during this time. The two liquid phases are recovered at the far end of the settler via overflow (organic) and underflow (aqueous) weirs.

Ideally the conditions within a settler will enable complete separation of the organic and aqueous phases, although rarely if ever does this occur. Whereas some process parameters affecting settler behaviour such as throughput rates can be readily varied, others such as the dimensions of a constructed settler cannot. In attempting to maximise phase separation, one very important aspect to consider is optimising the residence time available to all liquid molecules in the settler. This is best achieved by having a flow pattern close to plug flow in the settler. This is because, for a given average settler residence time, the benefit of increased residence and thus disentrainment time for some molecules in a non-plug flow system is outweighed by the detrimental effect of necessarily decreased residence time for other molecules ('short-circuiting'). Flow patterns and disentrainment behaviour in settlers can be affected by a variety of factors. For example, improvements in settler performance can be achieved by minimising the velocity differential between the organic and aqueous phases, improving the inlet feed distribution from the mixer box to the settler by eliminating reverse flows and macro eddies, improved coalescence with the aid of settler internals or 'furniture' like picket fences and coalescence packs currently used in the SX industry as well as preferential design and placement of furniture in areas of deep emulsion bands (Miller, 2006; Poulter et al., 2011). Measurement of fluid flow patterns within large scale process vessels like SX settler units is therefore an important step in understanding the effect of operating conditions and equipment design (e.g. picket fences) on flow behaviour in a settler and provides the opportunity to test the effectiveness of design changes and to optimise operating performance.

1.3. Computational modelling of liquid flow Patterns in SX Settlers

Computational modelling of various phenomena including liquid flow patterns in SX settlers has become more prevalent with the advent of more powerful computers over the past decade or so. This computational fluid dynamics (CFD) modelling provides a relatively effective way of examining computationally derived flow patterns for a given settler design, and assessing the impact of design changes on the resulting flow patterns. CFD typically arrives at a solution by solving partial differential equations for mass, momentum and turbulence and iterating it to the lowest possible errors. Miller (2006) provided a good insight into the CFD generated flow patterns within a settler. Kankaanpaa (2005) through his CFD studies showed that in the absence of picket fences, the inlet jet from the mixer to the settler protruded for long distances into the settler while a shallow inlet depth results in reversed flows of the aqueous phase from half way down the settler back to the inlet. Through its CSXT (Customised Solvent Extraction Technology) programme, Hatch has been using CFD as a design tool to develop and implement alternative inlet launder design called the smooth flow solvent extraction feed launder, CSXT feed distribution array as well as the CSXT bull nose phase splitter with the aim of improving the flow pattern and having effective separation between the phases (Poulter et al., 2011). However, it should never be forgotten that CFD is simply portraying a mathematical model of what is predicted to occur in reality. Ultimately, experimental validation of CFD-determined outcomes greatly increases the value of a particular CFD model as a tool. As such, reliable data from an operating site is required.

1.4. Liquid flow measurement using ultrasonic velocity profiling

The UVP technique employs an ultrasonic Doppler method to obtain a one-dimensional velocity profile along its beam path from the reflected echoes of tracer particles present within the measurement medium. Traditionally, this technique was developed to measure the profile of blood flow in a blood vessel (Satomura, 1959). It employs the 'pulsed echo' method, detecting the Doppler shift in the echo as a function of time after pulse emission and reflection of the ultrasound that comes from the interface of blood cells. Takeda (1986) first applied this technique to measure one-dimensional liquid velocities in Poiseuille and Taylor vortex flows. Since then numerous studies have been conducted to measure velocities of liquids like mercury (Takeda and Kikura, 2002), body lotion (Brunn et al., 2004), suspensions (Ouriev and Windhab, 2003; Xu and Aidun, 2005) as well as void fraction of bubbles in water (Murai et al., 2009).

UVP uses two fundamental principles to measure the velocity as well as the location of the fluid motion. A 'time-of-flight' technique is used to ascertain the spatial measurement location while the Doppler shift frequency is used to attain instantaneous flow velocity. For any measurement scenario using UVP, factors like selection of ultrasonic basic frequency, transducer settings and presence of tracer particles need to be carefully chosen. Position