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The application of deep eutectic solvent ionic liquids for environmentally-friendly dissolution and recovery of precious metals

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ABSTRACT

The processing of ore by hydrometallurgy or pyrometallurgy typically has a high energy demand, and associated release of carbon dioxide. Thus there is a need to develop more energy-efficient and environmentally-compatible processes. This article demonstrates that deep eutectic solvent (DES) ionic liquids provide one such method since they can be used to selectively dissolve and recover native gold and tellurium, sulphides and tellurides.

Ionic liquids are anhydrous salts that are liquid at low temperature. They are powerful solvents and electrolytes with potential for high selectivity in both dissolution and recovery. Deep eutectic solvents are a form of ionic liquid that are mixtures of salts such as choline chloride with hydrogen-bond donors such as urea. DESs are environmentally benign, yet chemically stable and, furthermore, the components are already produced in large quantities at comparable costs to conventional reagents.

Electrum, galena and chalcopyrite, as well as tellurobismuthite (Bi_2Te_3), were soluble in DES through an oxidative leach at 45–50 °C. Leaching rates determined by a novel technique employing an optical profiler were very favourable in comparison to the current industrial process of cyanidation. Pyrite was notably insoluble by an oxidative leach. However, pyrite, and indeed any other sulphide, could be selectively dissolved by electrolysis in a DES, thus suggesting a protocol whereby target inclusions could be liberated by electrolysis and then dissolved by subsequent oxidation.

Ionometallurgy could thus offer a new set of environmentally-benign process for metallurgy.

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1. Introduction

The extraction of metals from ores is often an energy-intensive process, requiring either smelting or leaching at elevated temperature, or the use of large quantities of strong acids or bases that require energy to produce. These processes often produce wastes, in gaseous, liquid or solid form, which require treatment before they can be safely disposed, and this waste treatment requires further energy. The energy input equates to a significant cost and an associated CO_2 footprint, both of which it would be desirable to reduce. Furthermore, some treatments, most notably cyanidation hydrometallurgy, has poor public perception and can negatively impact on the “social licence to operate” that all mining operations

must secure. The increasing intolerance to cyanidation has resulted in it being banned from use in a number of countries and states.

The question is whether the minerals processing community can develop new, low-energy, low-cost and more environmentally compatible metallurgical processes for the mining industry on a planet of over 9 billion people? The advent of heap-leaching and biohydrometallurgy have made significant advances in this direction, but there still remain many ores for which the most economic, or only, option is energy-intensive hydrometallurgical or pyrometallurgical treatments.

We propose that ionic liquids could provide new environmentally-friendly approaches to processing a variety of ores, in particular those that are difficult and energy-intensive to treat. There is now a significant literature and extensive application of these liquids in the recovery of metals from scrap and metal oxides (Abbott et al., 2011a,b), the processing of reactive metals (Vaughan and Dreisinger, 2008), as well as applications across a

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range of chemical processing (Wasserscheid and Welton, 2008). However, there has been only limited attention paid to the possibility of using ionic liquids in processing base and precious metal ores (Whitehead et al., 2004, 2007, 2009; Luczak et al., 2008; Dong et al., 2009). There is a vast array of different types of ionic liquids and many are unsuitable to large-scale applications such as mineral processing due to high cost, lack of availability or toxicity. In the following sections we explain what ionic liquids are, and then show that a group of ionic liquids known as deep eutectic solvents fulfil the requirements for industrial application. We then demonstrate that these ionic liquids can be used to rapidly dissolve a variety of ore minerals, including gold, at low temperatures, and show how the metal can then be recovered by electrolysis.

2. A new type of chemistry – ionic liquids

Ionic liquids are salts that are liquid at low temperature, typically <100 °C. These anhydrous liquids are composed of ions and, like high temperature molten salts, are electrolytes and powerful solvents. In contrast to aqueous liquids, where the solubility of metals is limited by the tendency for water to combine with metal ions and precipitate oxides and hydroxides, in water-free ionic liquids much higher metal concentrations can be achieved. High ligand concentrations are possible allowing much greater control on metal speciation in solution, whilst recovery from solution can be by electrowinning, cementation, ion exchange or precipitation (Abbott et al., 2011b). These features provide potential for high selectivity in both dissolution and recovery. Furthermore, ionic liquids have a wider electrochemical window than aqueous solutions and so can operate beyond the potential range of aqueous solutions making electrowinning of more reactive metals possible, whilst avoiding the evolution of gas at the electrodes (Smith et al., 2014). The higher metal concentrations that can be achieved, together with the fact that ionic liquids can be reused in a closed circuit, means that high volume/low concentration aqueous wastes are not produced.

In order to be suitable for large-scale metallurgical processing a reagent must be of low cost, readily available in large quantities, chemically stable to allow prolonged reuse, be environmentally benign, and have no restrictions (i.e. be a common, simple chemical with well-known characteristics). Unfortunately all the ionic liquids that have previously been tested for dissolution of precious or base metal ores (Whitehead et al., 2004, 2007, 2009; Luczak et al., 2008; Dong et al., 2009) fail to meet one or more of these criteria. Ensuring that all these industry-necessary criteria are met from the outset led to the development at the University of Leicester of the deep eutectic solvents (DESS). These are mixtures of salts, such as choline chloride, with hydrogen-bond donors such as urea or citric acid (Abbott et al., 2004). The eutectic mixtures have melting points that are significantly lower than their individual components, giving a room-temperature ionic liquid. It should be noted that DESSs are not an ionic liquid *sensu stricto*, since they are a mixture rather than a single salt, nevertheless they are anhydrous liquids made of ions and share many properties with ionic liquids *sensu stricto*, although also exhibit some significant differences (Smith et al., 2014). The solvent properties of DESSs can be adjusted by changing the hydrogen-bond donor, giving 10⁵ possible different liquids and allowing the possibility of tailoring the properties of the liquid to a specific process (Abbott et al., 2004). DESSs are environmentally benign, yet chemically stable. Their components are common, cheap chemicals, e.g. choline chloride (vitamin B4) is mainly used as an animal-feed additive, being already produced in large quantities (forecast to exceed 500 kt choline chloride p.a. in 2017; Global Industry Analysts, 2014) at low cost (~€2/kg). Urea is a common nitrogen fertilizer (2012 worldwide production capacity was ~184 Mt; Ceresana, 2012) that is non-flammable

and completely biodegradable, retailing at ~€0.5/kg. The behaviour of d-block metals and group IIIA–VA metals and semi-metals in DESSs is well-understood (Abbott et al., 2011b). It has previously been shown that metal oxide extraction by DESSs is economically viable even for low value metals (Abbott et al., 2009).

3. Demonstration 1 – dissolution rates of gold and other minerals in a deep eutectic solvent

Here we demonstrate how a variety of minerals can be dissolved from ore samples using iodine (I₂) as an oxidising agent within a DES. We show how the dissolution rates can be rapidly estimated using a micro-leach technique with an optical profiler, and compare these observed rates to those of existing techniques. We investigate a quartz vein-hosted orogenic-type gold ore with relatively small gold particles (90% <100 µm, most <40 µm; Dominy and Platten, 2008) because high efficiency of extraction of gold from such an ore would typically demand pyrometallurgy (roasting), a hydrometallurgical step (involving cyanidation), or both. In addition we investigate native tellurium and a telluride mineral, because telluride-bearing gold ores are typically refractory to cyanidation and are lost to tails (Spry et al., 2004; Zhang et al., 2010). Alternatively such ores may need to be roasted to make them amenable to cyanidation (Zhang et al., 2010). In either case tellurium (a rare element increasingly sought after for solar photovoltaic panel construction; Woodhouse et al., 2013) is lost and becomes a potential environmental contaminant. Thus approaches that might liberate and recover gold and valuable associated elements from ores could both increase the value gained from an ore and reduce environmental legacies.

3.1. Experimental

All samples were mounted in resin, cut and polished to produce a standard polished block with a flat surface as used for reflected light microscopy. The electrum, chalcopyrite, galena and pyrite investigated were all in two polished blocks of gold ore sample CGJ CON 002 from the Cononish gold deposit, Scotland, (Curtis et al., 1993; Spence-Jones, 2013; Hill, 2014) courtesy of Scotgold Resources Ltd. This gold-rich sample consists largely of pyrite, with lesser galena and chalcopyrite, in a quartz matrix. The electrum grain that was leached was silver-rich, measured as approximately 50 wt.% Au/(Au + Ag) by SEM-EDX.

Samples of native tellurium (BM 31691, Zlatna, Transylvania, Romania) and tellurobismuthite (Bi₂Te₃; BM 32070, Reed's Mine, Georgia, USA) were obtained as separate polished blocks from collections at the Natural History Museum, London.

The ionic liquid used was Ethaline, a DES mixture of 1 mol. eq. choline chloride and 2 mol. eq. ethylene glycol. Ethaline containing 0.1 mol dm⁻³ iodine was prepared following the procedure of Abbott et al. (2015). Polished blocks were etched by suspending them in a well-stirred solution at either 45 °C (tellurium, tellurobismuthite) or 50 °C (electrum, galena, chalcopyrite, pyrite). One mount of C/JG CON 002 was leached for 10 min after which the electrum and pyrite were measured. The other mount of C/JG CON 002 was leached for three 5-min leach steps and after each step the galena and chalcopyrite were measured and then returned to the solution without repolishing. The last step for galena etched into some inclusions and so has not been included. The tellurium and tellurobismuthite mounts were measured after leaching for 5 min, and then after a further 10 min. The tellurium was measured in two different locations on the mount. Ethaline was removed from samples by rinsing with water and then air drying prior to measuring etch depth.

2D and 3D optical images of the samples pre- and post-dissolution were captured on a Zeta Instruments Zeta 2000 optical

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