



## Short communication

## Applying electrohydrodynamic atomization to enhance mass transfer of metal salts from an aqueous phase towards ionic liquids

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## ABSTRACT

In this work a two coaxial nozzles configuration is used to investigate whether electro hydrodynamic atomization of an ionic liquid in combination with an aqueous metal salt solution could enhance the removal of metal salts. The technique was evaluated for the removal of manganese (II), cobalt (II) calcium and sodium chloride. Good metal salt extraction was observed for the water-presaturated ionic liquid, tetraoctylammonium oleate, at an applied electrical potential of 5 kV, which was slightly lower compared to mechanical mixing, but a higher separation factor was obtained between the transition metals and the alkali and earth alkali metals.

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## 1. Introduction

Heavy metals are a common source of pollution in water. Their origin is rather diverse. While arsenic (As) normally originates from the oxidation of sulfide minerals like arsenopyrite, lead (Pb) has its origin normally from old metallic distribution and drainage systems and mine machinery maintenance yards. Cadmium (Cd), zinc (Zn), manganese (Mn) and iron (Fe) have their source from industrial processes and mercury (Hg) is normally dispersed from small scale (artisanal) mining [1]. Among them Cd is especially known by its high toxicity and as an environmental hazard [2]. Nowadays, it is mainly As that has been mentioned as a potential health risk for drinking water in many countries [3–6].

Several techniques can be used to remove these metals from liquid effluents, e.g. precipitation, solvent extraction, ion exchange, adsorption, membrane filtration (micro, ultra, and nano), coagulation–flocculation, flotation and electrochemical methods [7]. Even though the list of suitable methods is large, the removal of such contaminants from wastewater streams below the standard

limits is still a challenge [8]. The main limitations of the current methods are the high capital and/or operational expenditure and the high standards for influent quality [9].

Among all current techniques, solvent extraction is commonly used for the separation and purification of metals [10]. It comprises basically a chemical extraction process, where an extractant agent is dispersed in a solution containing the contaminant using some form of energy, e.g. mixing or stirring. The removal process can be either an ion-exchange process, or liquid–liquid extraction with metal extractants. Examples of such extractants are di(2-ethylhexyl) phosphoric acid, tris(2-ethylhexyl)amine and liquid phosphine oxides dissolved in organic solvents such as kerosene or toluene [11,12]. This method is known by its high extraction efficiency, i.e. extraction efficiencies above 99% can be reached. However, it presents high operational costs due to the handling and disposal of the used solvents, which are normally highly toxic, flammable and/or volatile.

If assumed that the volatility of the organic solvents and the toxicity of the extractant and solvent are the main disadvantages of current chemical extraction processes, then finding “green” extractants”, i.e. bio-degradable/non-toxic extractants, would represent a big improvement for this industry. Recently, the use of Ionic Liquids (IL) has been considered as a potential solution

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[13–15].

ILs are known as substances entirely formed by anions and cations that melt at or below 100 °C [16,17]. The composition of an IL is a bulky organic cation combined with an inorganic or organic anion. The strong electrostatic interactions between the ions result in their characteristic properties, i.e. their negligibly low vapor pressure at room temperature and the high chemical and electrochemical stability [18]. Moreover, other properties like solubility and viscosity can be modified by substituting different anions and cations [19–24]. As well as their application, i.e. selecting ions with functionalities that interact with metals results in ILs suitable for metal salt extraction [25–27].

Currently, metal salt extraction with ILs is carried out using a mixer-settler configuration. This means that mechanical mixing of the IL with the aqueous metal solution is performed to enhance the contact between the extract and the solution containing the contaminant [28]. In practice, any other technique which could create an enhanced interface with less energy would improve the system's efficiency. A good possibility is to modify this interface using electrical forces, i.e. exposing the liquids to a strong electric field.

A technique that uses such principle is electrohydrodynamic atomization (EHDA) [29–32]. EHDA, or shortly electrospraying, is an atomization process, which implements electric stresses into the liquid leaving the nozzle. These stresses are caused by creating a strong electric field in the breakup region by applying an electric potential between the nozzle and a counter electrode. For a certain nozzle-counter electrode configuration (defining the electrical field strength) and a specified liquid, different spraying modes can be obtained depending on the applied potential difference, and the liquid flow rate. This paper only considers low flow rates. For low flow rates ( $\mu\text{L/h}$  –  $\text{mL/h}$ ) the mode changes by increasing the potential from dripping via intermittent to cone-jet mode (Fig. 3). By further increasing the potential a multi-jet mode appears. If the electric field strength becomes too high, sparks can take place. In the cone-jet mode the shape of the liquid surface is transformed into a cone and from the apex of the cone a liquid jet emerges, which is much thinner than the nozzle diameter. The jet then breaks up in small, highly charged droplets ( $\text{nm}$ – $\mu\text{m}$ ), which can be monodisperse. Among many other applications, electrospraying is used for drug delivery [33,34], in greenhouses [35], and for controlled deposition [36].

In EHDA the electric field is created by establishing an electric potential difference ( $\phi$ ) between the nozzle and a counter electrode placed at a certain distance from the nozzle's tip. Various nozzle/counter electrode configurations can be used. The most known one is the nozzle/plate configuration, in which the counter electrode is a metal plate placed below the nozzle tip [36]. Alternatively, configurations like coaxial cylindrical nozzles [37] and nozzle/ring [35] have also been successfully applied.

For a liquid with a high surface tension, such as water, the electric field necessary to overcome the surface tension stress and to transform the liquid surface into a stable one, is so high that sparking happens before the cone formation can be reached. A possible way to overcome this problem is to use two coaxial nozzles in where the outer liquid is a well electrosprayable liquid and the inner one is the non-electrosprayable liquid [38]. This process is also known as “electro-coextrusion” [39]. Literature also shows that certain ILs can be electrosprayed in a stable cone-jet mode [40]. If this is done with a two coaxial nozzle configuration, then also an extended contact surface between the two liquids can be realized.

In this work, a system with two coaxial nozzles was developed so that IL and salt solution can be simultaneously electrosprayed in the cone-jet mode, as shown in Fig. 1. The combination of EHDA with ILs was investigated for the first time to see whether it can be

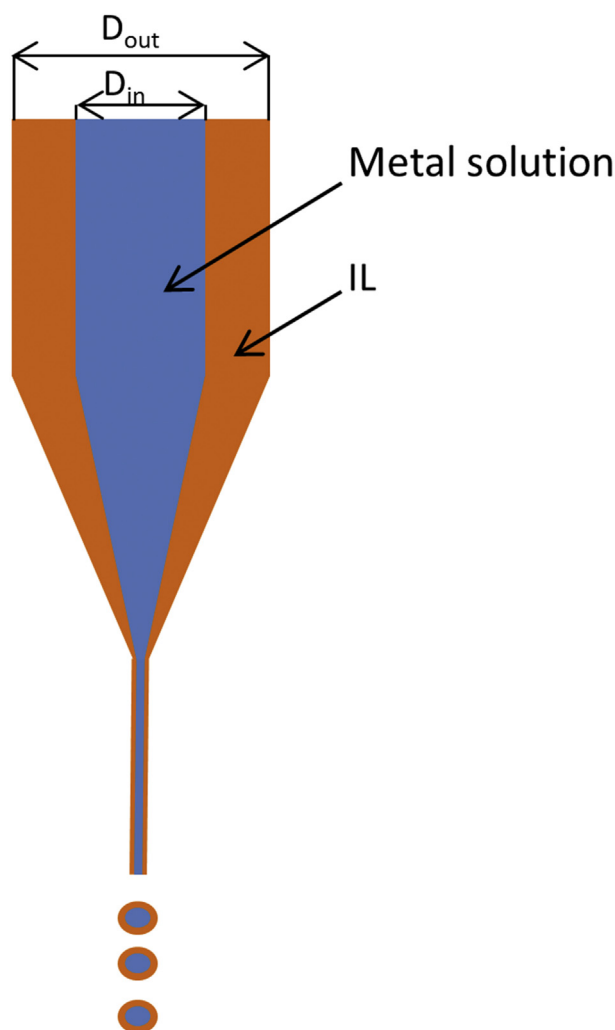


Fig. 1. Ideal cone-jet mode with the formation of encapsulated droplets.

used to improve the selectivity and extraction efficiency of metals into the IL solution.

## 2. Experiment and materials

### 2.1. Materials

The atomized liquids were an aqueous salt solution and the IL, i.e. tetraoctylammonium oleate ( $[\text{N}_{888}][\text{C}18:1]$ ). The IL was selected because it is already known as an efficient selective metal extractant for transition metals from light metals. As reported in the literature such mechanism is based on ion-pair extraction [25,41,42]. Before using the IL, it was pre-saturated with water to decrease the viscosity and to facilitate electrospraying. The aqueous metal salt solution was prepared by dissolving 0.5 g of each salt ( $\text{MnCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NaCl}$ ,  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) in demineralized water. All chemicals were purchased from Sigma–Aldrich. MilliQ water ( $\geq 18.2 \text{ M}\Omega \text{ cm}$ ) used throughout the synthesis was obtained by a Millipore Milli-Q® biocel, which used a Q-grade® column. The IL used in the experiments was synthesized using the same method as previously described and had a purity of  $\geq 95\%$  measured by  $^1\text{H}$  NMR [25].

The physical properties of both the IL and the aqueous metal salt solution were determined. (see Table 1) Density and viscosity were

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