



# Continuous-flow leaching in a rotating coiled column for studies on the mobility of toxic elements in dust samples collected near a metallurgic plant



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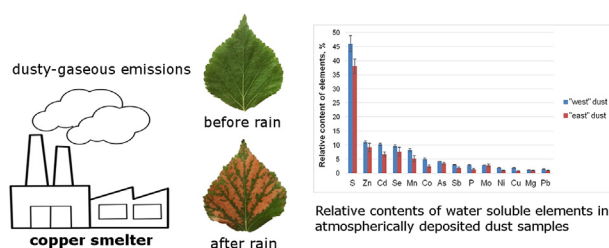
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## HIGHLIGHTS

- Dynamic leaching was applied to studies on the mobility of toxic elements in dust.
- Atmospherically deposited dust samples collected near a copper smelter were studied.
- Water and simulated "acid rain" were used as eluents.
- Up to 1.5, 4.1, 1.9, 11, and 46% of Pb, As, Cu, Zn, and S can be mobilized by water.
- Simulated acid rain results in higher recoveries of elements, except Cu and Pb.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Article history:

Received 22 October 2015

Received in revised form

26 November 2015

Accepted 30 November 2015

Available online 29 December 2015

Handling editor: Ralf Ebinghaus

### Keywords:

Atmospherically deposited dust

Copper smelter emissions

Toxic elements

Dynamic leaching

Rotating coiled column

Acid rain

## ABSTRACT

Continuous-flow (dynamic) leaching in a rotating coiled column has been applied to studies on the mobility of Zn, Cd, Cu, Pb, Ni, Sb, As, S, and other potentially toxic elements in atmospherically deposited dust samples collected near a large copper smelter (Chelyabinsk region, Russia). Water and simulated "acid rain" (pH 4) were used as eluents. The technique enables not only the fast and efficient leaching of elements but as well time-resolved studies on the mobilization of heavy metals, sulphur, and arsenic in environmentally relevant forms to be made. It is shown that up to 1.5, 4.1, 1.9, 11.1, and 46.1% of Pb, As, Cu, Zn, and S, correspondingly, can be easily mobilized by water. Taking into consideration that the total concentrations of these elements in the samples under investigation are surprisingly high and vary in the range from 2.7 g/kg (for arsenic) to 15.5 g/kg (for sulphur), the environmental impact of the dust may be dramatic. The simulated acid rain results in somewhat higher recoveries of elements, except Cu and Pb. The proposed approach and the data obtained can very useful for the risk assessment related to the mobility of potentially toxic elements and their inclusion in the biogeochemical cycle.

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## 1. Introduction

The industrial development in the South Ural, Russia has caused a tremendous damage to the environment. A large contaminated

area has been formed around the city of Karabash (Chelyabinsk region) where a copper smelter has utilized no treatment facilities for about one century. Recent renovation and modernization of the plant has considerably reduced dusty-gaseous emissions which still present the main factor of the anthropogenic load on the environment. Emissions of sulphur dioxide, Pb, Zn, Cd, Cu, and other chalcophile elements as finely dispersed aerosols result in the precipitation of “acid rains” (pH 3.5–4.0) characterized by anomalously high concentrations of metals in dissolved and solid phase forms. Concentrations of metals in these precipitates may be 2–3 orders of magnitude higher than in background ones (Gashkina et al., 2015). Hence, studies on the properties and chemical composition of atmospherically deposited dust in the area of Karabash copper smelter is of great importance for risk assessment related to the migration of toxic elements and their inclusion in the biogeochemical cycle.

Nowadays, it is well known that the impact of anthropogenic metal/metalloid ions on the environment cannot be evaluated by measuring the total concentration of individual trace elements (TE), because the mobility, bioaccessibility and, consequently, toxicity strongly depend on their chemical forms and type of binding. The identification and quantification of TE associated with predefined phases or soil compartments is defined as “fractionation analysis” according to the IUPAC recommendation (Templeton et al., 2000). Aqueous solutions of mineral acids, salts, oxidizing, reducing, and complexing reagents used as single extractants or in sequential extraction procedures are intended to simulate conditions whereby metals associated with certain mineralogical phases can be released. In general, sequential extraction may provide relevant information on the origin, physicochemical mobility, and transport of TE. Actually, the “nominal forms”, determined by operational fractionation, assist in the estimation of the amounts of elements in different reservoirs that can be mobilized under changes of the environmental conditions (Davidson et al., 1998). Since the 1970s a considerable number of sequential extraction procedures have been proposed for determining the forms of trace metals (Filgueiras et al., 2002; Gleyzes et al., 2002; Hlavay et al., 2004) and metalloids (Gleyzes et al., 2002) in soil, sediment, ash, and dust samples. Single medium extraction procedures employing diluted salt or acid solutions have also gained acceptance and can be regarded as a suitable method for predicting the plant uptake of TE (Fedotov et al., 2012).

Even though batchwise extraction methods have been consolidated as tools for investigation of the environmental behaviour of potentially toxic compounds, the significance of the results in terms of environmental availability is, in fact, debatable because naturally occurring processes are always dynamic, while the manual protocols, intended to simulate environmental scenarios, inherently are based on the establishment of a single equilibrium between solid and liquid phases. In recent years alternative methodologies relying on the continuous provision of fresh extractant volumes to the solid sample under investigation have been developed (Miró et al., 2005; Fedotov and Miró, 2008).

The application of microcolumndruges or microcolumns in the conduits of flow injection systems has attracted a particular interest for dynamic leaching studies as well as for TE partitioning and release rate investigations. Solutions of different extractants are sequentially pumped through a microcolumn packed with a dried solid sample (typically 10–80 mg). The technique enables to perform the continuous-flow leaching with on-line determination of elements in the effluent by inductively coupled plasma mass spectrometry (ICP-MS) (Beauchemin et al., 2002; Jimoh et al., 2004, 2005; Buanuam et al., 2006; Silva et al., 2007), atomic absorption spectrometry (Miró and Hansen, 2005) or hydride generation-atomic fluorescence spectrometry (Dong and Yan, 2005). Hence,

it becomes possible to access the mobility and fractionation of elements by real-time following the leaching process.

It has been also demonstrated that rotating coiled columns (RCC) can be used for continuous-flow (dynamic) leaching of trace metals (Fedotov et al., 2002, 2005a,b, 2006, 2007) and metalloids (Fedotov et al., 2005b; Savonina et al., 2012a; Savonina et al., 2012b) from contaminated soils, lake and river sediments. The solid sample (typically 0.5 g) is retained as a stationary phase in the rotating column, whereas leaching solutions of mineral acids, electrolytes (salts), and complexing agents are successively pumped through. A flow-through hyphenated analytical method has been proposed that enables not only the accelerated and efficient fractionation of trace elements species in environmental solids to be achieved but allows real-time studies on the leaching process to be made. Elements were determined in the effluent of RCC on-line by inductively coupled plasma atomic emission spectrometry (ICP-AES) (Fedotov et al., 2006, 2007).

Moreover, the microcolumn (MC) extraction and rotating coiled column (RCC) extraction have been comparatively studied (Savonina et al., 2012a, 2012b; Rosende et al., 2009) and the first attempt for harmonization of flow-through dynamic leaching using two methods has been made (Rosende et al., 2009). Similarities and discrepancies between both dynamic approaches were ascertained by fractionation of trace metals in certified reference materials, namely, SRM 2711 Montana Soil and GBW 07311 sediment, and two real soil samples as well. Notwithstanding the different extraction conditions set by both methods, similar trends of metal distribution were in generally found. The most critical parameters for reliable assessment of mobilisable pools of trace metals in worse-case scenarios are the size-distribution of sample particles, the density of particles, the content of organic matter and the concentration of major elements. For reference materials and a soil rich in organic matter, the extraction in RCC results in slightly higher recoveries of environmentally relevant fractions of TE, whereas MC leaching is more effective for calcareous soils (Rosende et al., 2009). It should be noted that in MC, the extractant flows through a packed particulate matter. Preferential flow channels for the leachant may be formed, thus lowering the effective contact surface between the samples and leaching solution. In RCC, particles may be either fixed to a given position at the column walls or migrate within the column thereby increasing the effective surface area. Under natural conditions, when rain falls, a particulate matter (especially dust) can be mobilized and entrained by water flow. Similar processes may occur in RCC. In general, however, both MC and RCC extractions look promising and can be successfully applied to environmental studies (Savonina et al., 2012a, 2012b; Rosende et al., 2009).

In our preliminary work, the continuous-flow leaching in RCC using water and simulated acid rain has been applied to studies on the mobility of trace elements in soil and sediment samples influenced by dumping of residues of the flood in the Mulde river region (Germany) in 2002 (Schreiber et al., 2005). Dynamic and traditional batch procedures were compared. It has been shown that the aqueous elution under RCC conditions is much more effective for the mobilization of heavy metals, as compared to the batch extraction. In this work the proposed approach has been extended to studies on the fractionation and mobility of heavy metals, arsenic, and other elements in quite specific samples of contaminated dust collected near a large copper smelter.

## 2. Material and methods

### 2.1. Samples and reagents

Atmospherically deposited samples of dust were collected using a brush and a plastic scoop on August 2013 at times of dry and

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