

Contents lists available at ScienceDirect

## Journal of Geochemical Exploration



journal homepage: <www.elsevier.com/locate/gexplo>

# Assessment of metals behaviour in industrial soil using sequential extraction, multivariable analysis and a geostatistical approach



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### article info abstract

Article history: Received 1 April 2016 Revised 17 October 2016 Accepted 31 October 2016 Available online 02 November 2016

Keywords: Chemical speciation Heavy metals Distribution maps Soil pollution

The main objectives of this study were to evaluate the degree of Pb, Cu, Zn, Cd, Cr and Ni pollution using pollution indexes and geostatistical approach, and to assess metals dynamic using sequential extraction procedure and multivariable statistical analysis in surface soils and soil profiles from an industrial complex as a base for a correct management in order to avoid health and environmental problems. Results showed that the industrial activity increases both Pb (103 mg kg<sup>-1</sup>), Zn (526 mg kg<sup>-1</sup>) and Cu (39 mg kg<sup>-1</sup>) concentrations and salinity in soil. Pollution indexes showed that industrial soils were moderately contaminated by Zn, Pb, Cu, Cr and Ni as well as a moderate ecological risk was reported. Two main areas were identified: southeast area with the highest metal concentrations, and northwest area with the lowest levels. Chemical speciation of metals showed that the residual phase was the dominant phase for all metals. However, Pb and Zn were highly associated to the reducible phase (25–30% and 35–40% respectively) and a significant concentration was associated to carbonates (5% for both metals). In contrast, Cu, Cr and Ni were mainly bound to the residual phase (>80% for all metals) with low concentrations retained to reducible phase, and very low concentrations bound to the most labile phases. Cd was the most mobile metal with high concentration associated to exchangeable (5%) and carbonates (15– 20%) phases. Therefore, Pb, Zn and Cd represent the greatest risk for human health and the environment. © 2016 Published by Elsevier B.V.

### 1. Introduction

The development of industrialization has caused an increase of soil pollution by metals in the last decades, whose effects are not only limited to industrial soils but also affect to the adjacent lands, which is especially intense when soils are used for discharge poorly treated liquid effluents and disposal of solid waste as well as deposition of exhaust gas from the industries [\(Fakayode and Onianwa, 2002; Wu et al.,](#page--1-0) [2011\)](#page--1-0). However the persistence and distribution of metals in industrial areas is mainly affected by factors such as the nature of parental material, properties of soil or climatic condition [\(Krishna and Govil, 2008](#page--1-0)). Metals in soil can remain for a long period of time acting as source and sink of contamination ([Acosta et al., 2015\)](#page--1-0) and therefore they are potentially hazard to ecosystems and human health [\(Yaylali-Abanuz,](#page--1-0) [2011\)](#page--1-0). Human exposure to metal pollutants may cause nervous, renal, cardiovascular and reproductive systems [\(Christoforidis and Stamatis,](#page--1-0) [2009\)](#page--1-0). Therefore, in order to develop policies on pollution control and environmental management, as well as, to make decisions on soil remediation procedures, it is necessary to carefully evaluate the behaviour and distribution of metals in soils.

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The contribution of metals to environmental pollution from industrial emissions has been the main subject of many studies in the recent years [\(Ordoñez et al., 2003; Al-Khashman and Shawabkeh, 2006;](#page--1-0) [Sekhar et al., 2006; Govil et al., 2008; Krishna and Govil, 2008; Wu et](#page--1-0) [al., 2011](#page--1-0)), where different indexes, such as contamination factor, enrichment factor or ecological risk index, have been used for determining the degree of soil pollution [\(Loska et al., 2004; Yaylali-abanuz, 2011;](#page--1-0) [Yuan et al., 2014](#page--1-0)). However, although the total metal content in soil is useful to estimate the degree of soil pollution, the mobility of metal does not necessarily depend directly on its total metal content ([Kumar](#page--1-0) [et al., 2013\)](#page--1-0). Chemical speciation is usually used to understand the geochemical processes governing metal mobilization [\(Yuan et al., 2004](#page--1-0)) because provide knowledge about the identification and quantification of different species, forms or phases of metals present in a material, such as soil, sediment or waste [\(Naji et al., 2010\)](#page--1-0), and its availability on the environment.

In addition, the identification of pollution sources, the risk areas, and the evaluation of metals behaviour in soil have been identified as key points to reach effective soil reclamations [\(Chen et al., 2009; Acosta et](#page--1-0) [al., 2011\)](#page--1-0). The use of multivariate statistical analysis combined with geostatistical techniques have been successfully applied in the identification of sources, evaluation of metal behaviour in soil and mapping of metal spatial distribution ([Rodríguez-Martín et al., 2006; Saby et al.,](#page--1-0) [2009; Maas et al., 2010; Lu et al., 2012; Li et al., 2013b; Shao et al.,](#page--1-0) [2014](#page--1-0)), being useful tools for developing future reclamation plans in

industrial areas and its surrounding. However, the use of advanced analytical and statistical methods combined with spatial distribution analysis for evaluating the risk posed at the specific site has been little studied.

The main objective of this study was to demonstrate that the combination of different techniques and methods (sequential extraction procedure, multivariable statistical analysis and geostatistical approach) is a powerful tool for an integral evaluation of the environmental risk in areas affected by metals. To reach this objective, the degree of metals pollution (Pb, Cu, Zn, Cd, Cr and Ni) and the delimitation of risk areas using pollution indexes and a geostatistical approach were carried out. Otherwise, the identification of sources of metals and their behaviour were evaluated using a sequential extraction procedure and a multivariable statistical analysis.

### 2. Material and methods

### 2.1. Study area and sampling

In order to evaluate the combination of analytical, statistical and geostatistical techniques for assessing contaminated sites, the most important industrial complex of Murcia city was selected (southeast of Spain), which is located in the southwest of the city. About 841 companies are situated in this industrial area, such as manufacturing paints,

steel product, chemicals, electrical materials, etc. The climate of the area is semiarid Mediterranean with an annual average temperature of 18 °C and annual precipitation of 350 mm. The geology of the area is dominated by Quaternary alluvial deposits from the Segura and Guadalentín rivers ([IGME, 1976](#page--1-0)). In the last decades, the soils where the industrial area is currently located were subjected to intense agricultural cultivation of lemon, oranges, cereals and vegetables. Currently, the industrial soil is classified as Calcaric regosol [\(W.R.B., 2014](#page--1-0)), characterized by low differentiation among horizons, a low organic matter content and high carbonates content. The study area also includes a natural area, without any influence of the industrial activities and with the same geological material. According with [W.R.B. \(2014\)](#page--1-0) the natural soil is classified as Haplic calcisol, characterized by the presence of calcic horizon, with low organic matter content. This area was used as control site to determine the pollution degree of the industrial soils.

The industrial area has a surface of 3  $km<sup>2</sup>$  where 10 surface soil samples (0–5 cm) were collected (Fig. 1). In addition, another 10 soil samples from the natural area were also sampled. At each sampling point, three sub-samples were collected 1 m apart from the vertices of a triangle to form a composite sample. Soil samples were taken using a soil spade. Two soil profiles were identified in the north-west and southeast of the industrial area corresponding to low and high pollution respectively (Fig. 1); four samples were collected from different depths (0–5 cm, 5–10 cm, 10–15 cm and 15–30 cm). All samples were taken



Fig. 1. Sampling location.

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