



# Trace element levels in an area impacted by old mining operations and their relationship with beehive products



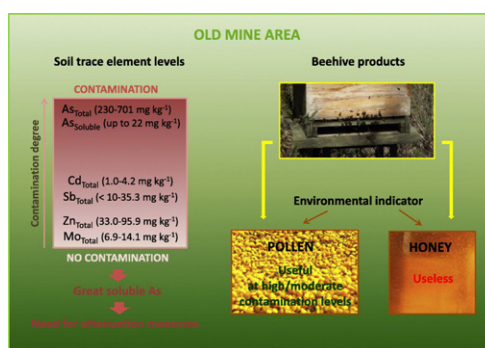
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## HIGHLIGHTS

- Areas impacted by past mining activities could entail serious environmental threats.
- The environmental status of a Roman mining area was assessed by litho-bio-sphere data.
- Analysis of soil (total/mobile/mobilizable) and honey/pollen trace element contents
- High potential risks derived from great soil mobile and mobilizable As contents.
- Pollen could act as environmental indicator at moderate/high contamination levels.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The environmental status of an area impacted by Roman mining activities was assessed in order to establish the current risks posed by such old mine emplacements. For this purpose, soil samples were collected throughout the mining area and analysed for their total, mobile and mobilizable trace element (As, Cd, Mo, Sb and Zn) contents. Additionally, beehive products (honey and pollen) were also sampled and evaluated for their use as environmental indicators of the area. The results obtained were compared with those from a control non-polluted area. The mine soils presented slightly increased levels of Cd and Sb (about 2- to 3-fold their normal soil concentrations), whereas the enrichment of As reached considerable levels, with concentrations almost ten-fold of those considered the threshold for causing toxicity. Leachable As contents exhibited very high values (1.2–21.9 mg kg<sup>-1</sup>), indicating the need for risk attenuation measures. All trace elements were mainly partitioned in the soil residual fraction, especially Mo (76–99%) and Sb (61–91%). Significant partitioning levels were also found in the reducible fraction of As (up to 35%) and Cd (up to 38%), and in the oxidizable fraction of Mo (up to 23%). The reducible pool of As was particularly relevant due to the eventual mobilization of this element under reducing conditions. Among the beehive products tested, honey proved not to be useful as an environmental indicator, whereas pollen showed great potential as an indicator when the contamination levels were moderate to high.

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## 1. Introduction

Increased trace element levels in soils can present a serious threat to the entire surrounding ecosystem. Although both natural and anthropogenic sources of trace elements can cause soil contamination, the latter usually causes the most severe impact on the environment and/or

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human health. This has been attributed to the increased input of trace elements into soils due to anthropogenic sources and to the greater availability of trace elements derived from these sources (Kabata-Pendias and Pendias, 1992; Kabata-Pendias and Mukherjee, 2007). Mining operations, industrial activities (smelting, power generation, electronics, ceramic and glass manufacture, chemical industries, etc.), waste disposal and agricultural practices are among the main human activities known to cause soil pollution with trace elements (Alloway, 1995; Kabata-Pendias and Mukherjee, 2007). Of these, mining and the processing of mineral ores are considered the earliest anthropogenic source of trace elements, going back as far as the period between the Bronze Age and Roman times (Ernst, 1998; Kabata-Pendias and Mukherjee, 2007). As a result, this activity has caused global, regional and local enrichment of soils with diverse trace elements (Ernst, 1998). Likewise, the mines in operation during the nineteenth and early twentieth century generated huge amounts of waste materials containing high levels of trace elements (Alloway, 1995). The poor management of these waste products facilitated the dispersal of trace elements through leaching and run-off processes, and by the mechanical transport of these particles. Consequently, these mine-impacted areas can be severely polluted with different trace elements, where the most common are As, Cd, Cr, Cu, Hg, Ni, Pb, Sb, Tl and Zn. All of these are considered as priority pollutants by the U.S. Environmental Protection Agency (EPA). Except for Cu and Zn, these trace elements are highly toxic to mammals and cause medium to high phytotoxic effects (Kabata-Pendias and Mukherjee, 2007). The need for the environmental evaluation and the control of mine emplacements to avoid the spread of further pollution is considered within national and international environmental strategies world-wide.

Different approaches can be used to carry out the environmental assessment of such areas, including the strict sampling and analysis of impacted environmental compartments or the use of certain organisms or materials that can indicate the levels of pollution. Among the different proposed indicators, honeybees and beehive products are considered useful tools for the global monitoring of different environments (Leita et al., 1996). Trace elements can be transferred to honeybees from all of the environmental compartments (soil, vegetation, air and water) within the areas covered by forager honeybees (Leita et al., 1996). Usually, these insects actively forage within the 1.5 km range surrounding the hive (Perugini et al., 2011). Therefore, honeybees and beehive products supply integrated representative samples from a relatively wide area (Leita et al., 1996; van der Steen et al., 2012).

The main objective of this work was to assess the environmental status of a currently touristic and recreational area impacted by old mining operations using two different strategies: 1) soil sampling and analysis of total, mobile and mobilizable trace element contents, and 2) the evaluation of the use of beehive products (pollen and honey) as environmental indicators of the study area.

## 2. Materials and methods

### 2.1. Study area

The studied mine-impacted area is located about 2 km from the east of the village of Tapia de Casariego, in the north of the autonomous community of Asturias (Spain). Mining activities in this area are related to the exploitation of the Salave (gold) deposit. This deposit is located in the northern end of the Oscos belt, one of the four gold belts within the northwest of the Iberian Hercynian Massif. The Salave deposit is found in granodiorite, which is intensively affected by hydrothermal alterations. Gold mineralization occurs in fracture zones of a NE-SW direction, being mainly associated with arsenopyrite (FeAsS) and variable amounts of other sulphides such as pyrite (FeS<sub>2</sub>), stibine (Sb<sub>2</sub>S<sub>3</sub>) and sphalerite (ZnS) (Spiering et al., 2000; Rodríguez Terente, 2007). Another type of mineralization, mainly comprised of molybdenite (MoS<sub>2</sub>) together with minor bismuthinite (Bi<sub>2</sub>S<sub>3</sub>), also occurs in

this deposit in subvertical quartz veins. It is dimensionally much less important than that of gold (Rodríguez Terente, 2007).

The Salave gold deposit was exploited by the Romans in the first century CE by the “ruina montium” mining system that used hydraulic methods. Approximately 4–6 Mt of material was excavated with an estimated gold production of 5–7 t (Lavandeira, 1992; Lyndex, 1994; Rodríguez Terente, 2007). There are no records of further significant gold production after Roman times. However, during the last fifty years, an intensive gold exploration has been performed in the Salave deposit by several mining companies. Existing gold resources are estimated to be about 60 t, being one of the main gold deposits in Europe (Spiering et al., 2000). Even today, remnants of the past auriferous exploitation in the mining area are still recognizable (open pits, aqueducts, drainage tunnels, canals, etc.). Additionally, some exploitation of the Salave deposit for molybdenum took place around the middle of the past century, although this activity was minor.

### 2.2. Sampling

Soil sampling was performed within the area corresponding to the Roman exploitation and in a nearby area not directly affected by mining activities (Fig. 1). This latter area, situated approximately 3 km from the south of the mine, was used as a control. In both mining and control areas there were apiaries that belonged to the FAPAS pollination stations. Surface soil samples (0–10 cm) of about 2 kg were collected along a 500-m transect at distances of 10, 25, 50, 100, 250 and 500 m from the apiaries during the summer period of 2014 (Fig. 1). Beehive products, namely pollen and honey, were also collected from apiaries within the mine and control areas. These products were sampled during the spring-summer period of 2014. Pollen samples were collected at three different time points (mid-March, mid-June and mid-September) in pollen traps that were set over the entrance of the hive several days before sampling. Honey samples were collected during mid-September from honeycombs. The pollen samples collected in the mine area mostly belonged to the following plant species: *Eucalyptus* sp., *Salix* sp., *Brassicaceae*, *Cytisus scoparius* and *Taraxacum* sp. (mid-March sampling); *Ligustrum vulgare*, *Datura* sp., *Scabiosa* sp., *Reseda luteola*, *Plantago* sp., *Cytisus scoparius*, *Brassicaceae* and *Rubus ulmifolius* (mid-June sampling); *Cytisus scoparius*, *Trifolium repens*, *Allium* sp., *Datura* sp., *Hedera helix*, *Lotus creticus*, *Taraxacum* sp. and *Lythrum salicaria* (mid-September sampling). The pollen samples collected in the control area mostly belonged to the following plant species: *Eucalyptus* sp., *Cytisus scoparius*, *Taraxacum* sp., *Brassicaceae*, *Acacia* sp., *Prunus spinosa* and *Pinus* sp. (mid-March sampling); *Crataegus monogyna*, *Eucalyptus* sp., *Brassicaceae*, *Reseda luteola*, *Ligustrum vulgare*, *Taraxacum* sp. and *Allium* sp. (mid-June sampling); *Cytisus scoparius*, *Trifolium repens*, *Brassicaceae*, *Calluna vulgaris*, *Hedera helix* and *Taraxacum* sp. (mid-September sampling). The plant species identified in pollen samples correspond to plant species characteristic of each the particular study areas.

### 2.3. Sample characterization

Soil samples were subjected to physicochemical and environmental characterization after being air-dried and sieved through a 2-mm mesh sieve. The following soil physicochemical properties were determined in triplicate: pH by the saturated paste method, organic matter (OM) following the Walkley-Black method (Walkley, 1947), amorphous/low-crystalline Fe and Al oxides by extraction with ammonium oxalate under the darkness, crystalline Fe and Al oxides by extraction with ammonium oxalate + ascorbic acid according to the procedure of Wenzel et al. (2001), available P by the Olsen method (Olsen et al., 1954), and particle size distribution by the pipette method (Gee and Bauder, 1986). An acid soil, whose OM and available P contents were determined by an accredited laboratory, was used as a reference material to verify the accuracy of soil OM and P determinations. Ground white quartz samples, spiked with synthetic ferrihydrite or amorphous

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