



Research article

Sustainability of an *in situ* aided phytostabilisation on highly contaminated soils using fly ashes: Effects on the vertical distribution of physicochemical parameters and trace elements



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ABSTRACT

Aided phytostabilisation using trees and fly ashes is a promising technique which has shown its effectiveness in the management of highly metal-contaminated soils. However, this success is generally established based on topsoil physicochemical analysis and short-term experiments. This paper focuses on the long-term effects of the afforestation and two fly ashes (silico-aluminous and sulfo-calcic called FA1 and FA2, respectively) by assessing the integrity of fly ashes 10 years after their incorporation into the soil as well as the vertical distribution of the physicochemical parameters and trace elements (TEs) in the amended soils (F1 and F2) in comparison with a non-amended soil (R). Ten years after the soil treatment, the particle size distribution analysis between fly ashes and their corresponding masses (fly ash + soil particles) showed a loss or an agglomeration of finer particles. This evolution matches with the appearance of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) in FA2m instead of anhydrite (CaSO_4), which is the major compound of FA2. This finding corresponds well with the dissolution and the lixiviation of Ca, S and P included in FA2 along the F2 soil profile, generating an accumulation of these elements at 30 cm depth. However, no variation of TE contamination was found between 0 and 25 cm depth in F2 soil except for Cd. Conversely, Cd, Pb, Zn and Hg enrichment was observed at 25 cm depth in the F1 soil, whereas no enrichment was observed for As. The fly ashes studied, and notably FA2, were able to reduce Cd, Pb and Zn availability in soil and this capacity persists over the time despite their structural and chemical changes.

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1. Introduction

Since the first industrial revolution, constantly growing industrialisation and urbanisation as well as successive economic crises have profoundly affected the European landscape and soils. In 2011–2012, the European Environment Information and Observation Network for soil (EIONET-SOIL) identified approximately 342,000 contaminated sites at the European scale (Panagos et al., 2013). In France, 6002 contaminated or potentially contaminated sites are referenced in the Basol soil database (BASOL, 2015).

According to the Basol data, 64% of these sites are contaminated by trace elements (TEs) (i.e. As, Ba, Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Se, and Zn). Many sites are located in the North of France (663) in the former mining area where coal mining and smelting activities have widely contaminated the surrounding environment. This is the case around the former Metaleurop Nord lead smelter (located in northern France), which generated huge atmospheric emissions of dusts for more than a century, up to 2003 (Douay et al., 2006; Frangi and Richard, 1997). These dusts have led to high contamination of agricultural soils around the smelter. Main pollutants were Cd, Pb and Zn, and to a lesser extent Ag, As, Bi, Cu, Hg, In, Ni, Sb, Se, Sn and Tl (Sterckeman et al., 2000, 2002). Because of (i) the ubiquity of these pollutants, (ii) their environmental persistence and (iii) their hazardous effects on the environment and the human health, the

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management of TE-polluted soils is a major concern for all industrialised countries (Ali et al., 2013).

Since the early days of research on the remediation of TE-contaminated soils, various techniques have been assessed: physical (soil replacement, thermal desorption), chemical (leaching, fixation, electrokinetic, vitrification) and biological (plants, microorganisms, animals) techniques (Yao et al., 2012). It was shown that the application of any of these techniques depended on the actual or future soil uses, the level of TE contamination, the soil depth affected, the area of the contaminated zone and the economic constraints. In the case of a large area highly contaminated with TEs, biological and chemical immobilization appears to be the most suitable method to decrease metal mobility and bioavailability at the lowest cost (Mench et al., 2010). Indeed, TEs can be fixed by plants into the rhizosphere through adsorption and/or precipitation, thus limiting their migration into groundwater and the food chain (Ali et al., 2013). This technique, called phytostabilisation, offers the main advantages of being inexpensive, environmentally friendly, and conservative for soil quality (Salt et al., 1998). This technique can be used in combination with *in situ* metal immobilisation promoted by soil amendments. This remediation strategy is known as aided phytostabilisation (Alvarenga et al., 2009a). A decrease in the mobile TE pool in soil, promoted by amendments through sorption or precipitation reactions, allows the settlement and the growth of vegetation as well as ecosystem restoration on highly contaminated sites (Ruttens et al., 2006). Moreover, some of these amendments can improve the agronomic quality of soils (Ram and Masto, 2014; Sabir et al., 2015). This phytomanagement has been assessed using various amendments (poultry manure, iron grit, steel shot, fly ash, red mud, etc) and plant genera (*Agrostis*, *Populus*, *Miscanthus*, *Phaseolus*, *Panicum*, *Lolium*, etc.) (Alvarenga et al., 2009a, 2009b; Lee et al., 2014; Lopareva et al., 2011a; Marchand et al., 2011; Ruttens et al., 2006). These studies were often conducted using greenhouse experiments and to a lesser extent with a lysimeter (Houben et al., 2012; Kumpiene et al., 2011; Mench et al., 2003; Waterlot et al., 2011), but field trials are still too scarce (Wang et al., 2014).

To highlight the advantages of this management on a large scale, a field experiment was set up in 2000 on a former agricultural soil contaminated by the past atmospheric emissions of the former Metaleurop Nord smelter. This experimentation has already been used to assess the effects of aided phytostabilisation using fly ashes (FA), woody plants and herbaceous plants on the soil's physico-chemical parameters, the vegetation and the soil mesofauna (Demuyne et al., 2014; Grumiaux et al., 2010; Lopareva-Pohu et al., 2011a, 2011b; Pourrut et al., 2011). In 8 years, soil has undergone a structural and chemical transformation through the formation of organic layers, root system growth and mesofauna colonisation under variable climatic conditions. As in most studies, the experiments conducted to date on this experimental site have been restricted to the topsoil.

Although the aided phytostabilisation technique calls on slow chemical and biological processes and is sustainable over time, the composition and chemical properties of amendments over time as well as their long-term effectiveness and their impacts on the deeper layers of the soil are some of the important topics of phytomanagement that have not or only rarely been studied in the literature. The aim of this study was therefore to investigate i) the chemical and structural composition of fly ashes 10 years after their incorporation into the soil as well as their interactions with soil particles, ii) the distribution of certain chemical parameters as well as As, Cd, Hg, Pb and Zn concentrations along the soil profiles and iii) the amendment impacts on the mobility and availability of Cd, Pb and Zn along the top 30 cm of soil 10 years after the soil treatments.

2. Materials and methods

2.1. Experimental site description

The area impacted by the past activities of the lead smelter is a complex environment where the urban and farming uses are tightly interwoven. TE contamination of agricultural soils is limited to a ploughed layer at a depth of 25–30 cm (Sterckeman et al., 2000, 2002). The concentrations correspond to 1–50 times the concentrations measured on regional agricultural topsoil concentrations (Sterckeman et al., 2006).

The experimental site was described in detail in a previous paper which assessed the influence of fly ash aided phytostabilisation on topsoil parameters (Lopareva-Pohu et al., 2011b). Briefly, a former agricultural field, located 600 m from the former smelter, was divided into three plots, approximately 3000 m² each. In 2000, two of them (F1 and F2) were amended at a rate of 23.4 kg m² with silico-aluminous fly ashes (FA1) and sulfo-calcic fly ashes (FA2). The third (R) was not amended and was considered as the reference plot. Then 1800 trees were planted on the three plots following a regular scheme alternating five species (*Robinia pseudoacacia* L., *Alnus glutinosa* (L.) Gaertner, *Quercus robur* L., *Acer pseudoplatanus* L., *Salix alba* L.) as described by Lopareva-Pohu and collaborators (2011b).

2.2. Soil description

On each plot (R, F1, F2), a soil pit was manually dug. Particular attention was paid to the location of the three pits so that they would be surrounded by the same tree species. Soil profiles were described according to the FAO guidelines for soil description (2006).

2.3. Sampling and soil sample preparation

Soil samples were collected according to a systematic 5-cm depth increment from the topsoil down to a 55-cm depth. For each increment, soil samples were taken with a steel knife over the entire pit width. In the laboratory, samples were prepared according to the ISO 11464 standard. After root removal, each soil sample was dried at a temperature not exceeding 40 °C and then crushed to pass through a 2-mm sieve. Coarse materials were removed. An aliquot of this prepared soil sample was then milled to pass through a 250- μ m sieve with an ultracentrifuge mill (PM200, Retsch, Germany). During the soil profile description, masses of fly ashes were commonly observed in the organo-mineral horizon (A) of the F1 and F2 plots. These masses of about 1 cm³ are from silico-aluminous fly ashes FA1 (masses named FAm1) in the F1 plot and sulfo-calcic fly ash FA2 (masses named FAm2) in the F2 plot.

2.4. Analysis

2.4.1. Fly ashes

FA1 and FA2 ashes were first characterised by Lopareva-Pohu et al. (2011b). This analysis was completed by the determination of their particle size distribution, the pH and the total concentrations of Ca, P (expressed as P₂O₅), S, Al, Fe, As, Cd, Hg, Pb and Zn as well as their mineral composition.

The particle size distribution was determined by laser diffraction using a Beckman Coulter LS13320 analyser and considering an aqueous suspension (Hleis et al., 2013). The mineralogy was determined by X-ray diffraction (XRD) on a BRUKER D8 Advance diffractometer using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) in the 2θ range 10°–70°, with a step size of 0.02° and an integration time of 4 s (Hleis et al., 2013). The pH of FA1 and FA2 was determined using the

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