

POTENTIALLY TOXIC ELEMENTS IN ULTRAMAFIC ROCKS AND SOILS: A CASE STUDY FROM THE VOLTRI MASSIF (NW ITALY)

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INTRODUCTION

High concentrations of potentially toxic elements (PTEs) in surface and near-surface environment may be related both to anthropogenic, including industrial and agricultural activity, and geogenic source, mainly due to natural weathering of rocks.

Considering the geogenic sources, ultramafic rocks (*e.g.*, peridotite and serpentinites) are among the most critical from the environmental point of view; in fact, they are characterized by high contents of Cr, Ni, Co, and other PTEs, which can have potential harmful impact on ecosystems and human health if released into soils and waters during weathering and pedogenic processes (Brooks, 1987).

Despite a general similarity in the whole rock chemical composition, the concentration and distribution of PTEs in ultramafic rocks is highly variable and can range up to two orders of magnitude, mainly because of heterogeneities in mineralogy and mineral chemistry, texture, and structural properties of the ultramafic rocks (Kierczak *et al.*, 2016; Echevarria, 2018) due to the different geological settings, metamorphic overprint, and geodynamic evolutions (Deschamps *et al.*, 2013).

During the weathering of the ultramafic rocks, these substantial differences can directly affect soil chemistry, chroma, depth, and weathering intensity as well as the nature and the relative abundance of primary residual minerals and newly formed authigenic phases (Oze *et al.*, 2004; Chardot *et al.*, 2007; Alexander & DuShey, 2011; Kierczak *et al.*, 2016; Echevarria, 2018).

The soils developed on ultramafic bedrock play an important role in environmental management due to the high natural content of PTEs (such as Cr, Ni, and Co; Caillaud *et al.*, 2009) commonly exceeds, up to one order of magnitude, the concentration limits laid down by environmental agencies and governments (Kumarathilaka *et al.*, 2014; Tashakor *et al.*, 2014; Marescotti *et al.*, 2019). These extremely high PTEs concentrations (*e.g.*, Cr, Ni, and Co) are related to the abundance of PTEs-bearing minerals inherited by bedrocks and subordinate authigenic minerals.

These PTEs-bearing minerals lead to soil metal concentrations above the limits allowed by legislation, impeding, therefore, to discriminate the anthropogenic and natural sources using threshold concentrations when these minerals are present. Hence, one of the most complex problem to be faced for ultramafic soils is the determination of background levels and baseline values for PTEs.

The main objectives of this work were: *i*) to determine the mineralogy and the chemistry of PTEs of ultramafic rocks and soils; *ii*) to evaluate how lithological, textural, and structural properties of different ultramafic bedrocks with different degree of serpentization and deformation may affect the re-distribution and the fate of PTEs in ultramafic soils during pedogenesis; *iii*) to define the micromorphological, mineralogical, and chemical variations occurring in ultramafic soils, in order to understand the role of the primary and authigenic mineral phases on the occurrence and potential mobility of PTEs (particularly Cr, Ni, and Co); *iv*) to assess their environmental implications in the ecosystem and environmental management.

GEOLOGICAL BACKGROUND

The study area is located in the NW area of the Voltri Massif (VM), a wide meta-ophiolitic body with sedimentary cover at the southernmost termination of the Western Alps (Ligurian Alps, NW Italy; Fig. 1A).

The VM represents a remnant of the Jurassic Ligurian Tethys and is composed by tectono-metamorphic units accreted during the Late Mesozoic to Cenozoic Alpine orogenesis (Capponi & Crispini, 2008 and references therein). These units consist mainly of mafic to ultramafic rocks derived from different paleogeographic domains, including subcontinental mantle, oceanic lithosphere with sedimentary covers, and subordinate continental crust (Capponi *et al.*, 2016). The units of the VM are characterized by a polyphase tectono-metamorphic evolution and metamorphic imprint, from eclogite and blueschist facies prograde peak to retrograde greenschist facies (*e.g.*, Messiga *et al.*, 1983; Malatesta *et al.*, 2012; Capponi *et al.*, 2016; Scarsi *et al.*, 2018).

The study area comprises partially serpentinized spinel- and plagioclase-bearing lherzolite, with minor harzburgite, dunite lenses and pyroxenite bands, antigorite serpentinites and serpentine schists (Fig. 1B).

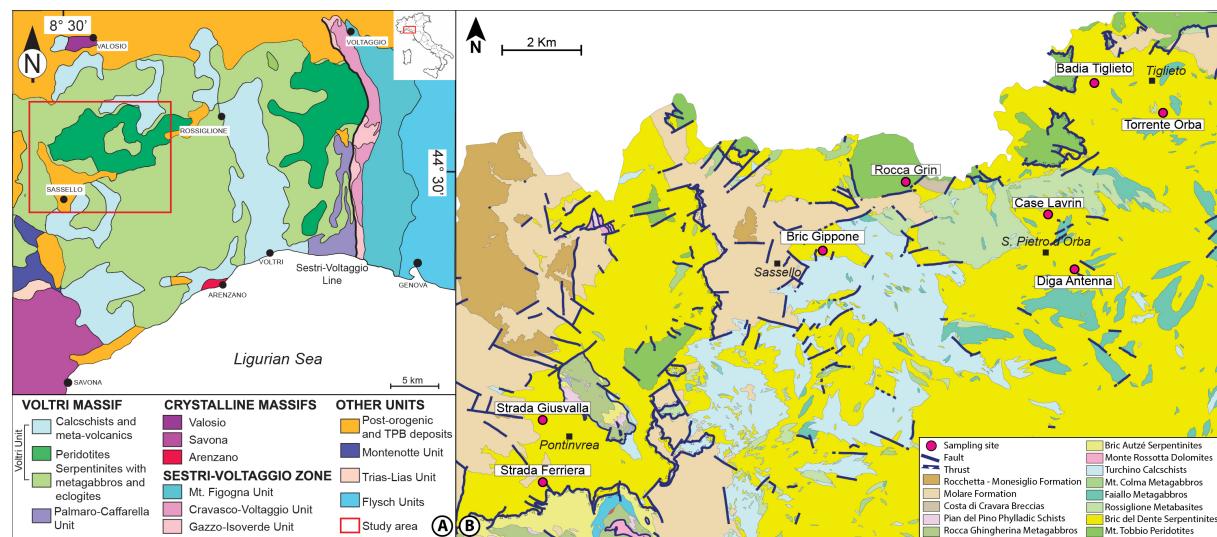


Fig. 1 - A) Geological map of the Voltri Massif and adjoining Units. Modified after Capponi & Crispini (2002). B) Geological map of the study area with the sampling sites location (red dots). Modified after Capponi *et al.*, 2013.

MATERIALS AND METHODS

A multiscale and multi-analytical approach was used in this work. *i)* A detailed field and geo-structural survey were performed in eight sites of the VM with good exposures of ultramafic rocks (*e.g.*, roadcut, abandoned quarries, stream banks), that were representative of significant structural and textural variations and degree of serpentinization. *ii)* Soil properties including color (Munsell® Soil Color Charts, 2000), grain-size (Folk, 1954), grain-morphology, texture, and structure were determined either in situ and in laboratory and used to distinguish the soil horizons as well as to group soils according to the FAO-WRB classification (IUSS, 2015). *iii)* Micromorphological analyses for qualitative weathering evaluation (Delvigne, 1998) and preliminary mineralogical/petrographic determination of rock and skeleton soil samples were carried out using polarized-light optical microscopy and SEM-EDS. *iv)* Qualitative and quantitative mineralogical analyses on samples were performed by means of XRPD; three different granulometric fraction (sand, silt, and clay fraction) of selected soil samples, were also analyzed by means of Synchrotron Radiation XRPD at the MCX beamline at the Elettra Synchrotron Radiation Facility (Trieste, Italy). Rietveld refinements were performed with General Structure Analysis Software (GSAS; Larson & Von Dreele, 2004), following the procedure described in Frondini *et al.* (2014). The composition of the clay fraction was analyzed at the Department of Physics and Geology at the University of Perugia after the separation and treatments procedure reported in Konta, (2001). A semi quantitative analysis of the mineralogical clay composition was performed using the “X’Pert Highscore” software. *v)* The mineral chemistry (major, minor, and trace elements) were determined either by EMPA-WDS (University of Milano, Italy) and in-situ Laser Ablation-ICP-MS (University of Perugia, Italy).

vi) Bulk chemical analyses were assessed by means ED-XRF (GeoSpectra s.r.l. - Spin-Off company of the University of Genova, Italy) and by ICP-AES (Regional Agency for Environmental Protection of Liguria, Genova, Italy).

RESULTS AND DISCUSSION

PTEs in the ultramafic rocks

The studied rocks are represented by ultramafic rocks with different serpentinization and deformation degree: *i*) partially serpentinized peridotite (PSP; Fig. 2A) with a serpentinization degree that ranges between 50 to 90%; *ii*) massive serpentinites (MS; Fig. 2B) with a prevailing undeformed massive texture, with a serpentinization degree that ranges between 90% to 100%; *iii*) foliated serpentinites (FS; Fig. 2C) with a serpentinization degree close to 100%.

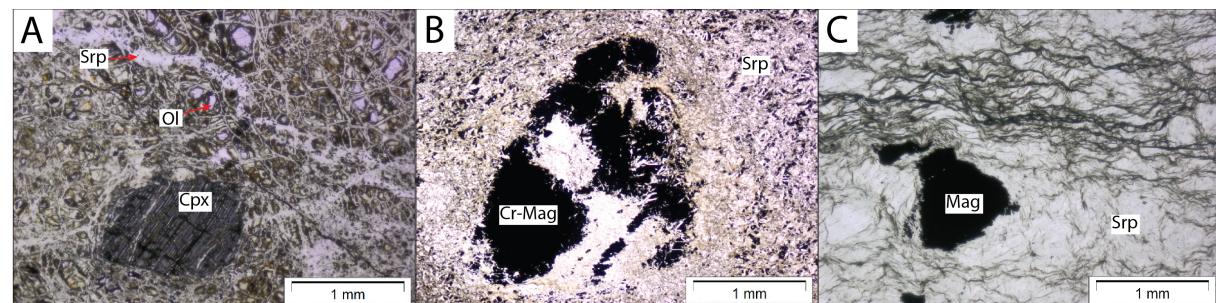


Fig. 2 - Photomicrographs of the studied ultramafic rocks: A) mesh texture with olivine relics and bastitic pyroxenes (PSP; plane-polarized light - PPL); B) magnetite and serpentine minerals aggregates within interpenetrating texture (MS; PPL); C) fine-grained magnetite-rich layers within serpentinite schist (FS; PPL). Mineral abbreviations after Whitney & Evans (2010). Modified from Fornasaro *et al.* (2019)

In the studied rocks, the main PTEs-bearing phases are represented by spinel-group minerals (Cr-spinel, Cr-magnetite, and magnetite) and subordinately by silicates (*i.e.*, serpentine minerals, olivine, pyroxenes, and chlorite) and weathering products (mainly Fe-oxides and -oxyhydroxides).

The highest concentrations of Cr are present in Cr-spinel (in PSP), ferrian chromite, Cr-magnetite (both in PSP and in MS), and magnetite (in all the three rock groups). Considerable amounts of Cr are also present in pyroxenes (up to 13000 ppm in enstatite), chlorites (up to 5920 ppm), and subordinately serpentine minerals (up to 9400 ppm in antigorite occurring in bastite textures).

The main Ni-bearing minerals are olivine (up to 3510 ppm) and subordinately serpentine minerals (up to 3200 ppm in antigorite occurring in the mesh textures), clinopyroxenes (up to 2200 ppm in diopside), chlorite (up to 1630 ppm), and talc (up to 1330 ppm). In the spinel-group minerals, Ni increase from Cr-spinels (Ni median value = 1480 ppm) to magnetite (Ni median value = 3130 ppm), according to the serpentinization degree.

The highest concentration of Co is present in olivine (up to 660 ppm), antigorite occurring in the mesh textures (up to 600), chlorite (up to 330 ppm), and talc (up to 380 ppm). In spinel-group minerals, Co increase from Cr-spinels (Co median value = 580 ppm) to magnetite (Co median value = 1030 ppm), *i.e.* with the increasing degree of serpentinization.

Enstatite and ilmenite are important carrier of V (up to 1790 ppm and 20390 ppm, respectively), whereas clinochlore (up to 730 ppm), talc (up to 1010 ppm), and ilmenite (up to 1880) show a high content of Zn. In spinel-group minerals V and Zn content decreases from Cr-spinel (up to V 1130 ppm, up to Zn 2680 ppm) toward magnetite (up to Zn 260 ppm, up to V 410 ppm).

The main authigenic phases forming by oxidation and weathering of primary minerals are Fe-oxides (hematite) and -oxyhydroxides (goethite) which occur as partial to complete replacement after magnetite or form cryptocrystalline aggregates within veins or in the surrounding oxidation halos. Although the chemical analyses of these minerals systematically refer to the bulk chemistry of the aggregates, the PTEs concentration of Fe-

oxides and oxyhydroxides is generally very high (Ni up to 51000 ppm, Cr up to 8370 ppm, Co up to 1920 ppm, and V up to 380 ppm).

Since the spinel-group minerals have the highest contents of PTEs among the mineral phases determined in this work, the trace element composition of these minerals was compared with the different textures, microstructures and degrees of serpentinization/deformation of the studied rocks. In the three rock types, the spinel-group minerals occur in three main classes: *i*) porphyroclasts (mainly Cr-spinel) with various degree of recrystallization (from ferrian chromite to magnetite); *ii*) spinels occurring within pseudomorphic and non-pseudomorphic texture (mainly magnetite); *iii*) spinels re-oriented along the foliation (mainly magnetite); *iv*) spinels within veins (magnetite)

Cr (up to 4180 ppm), Ni (up to 3900 ppm), and Co (up to 330 ppm), and subordinately Cu, V, and Zn are always present in significant concentrations (Fig. 3). Cr and Ni vary systematically with the rock types (*i.e.*, with the degree of serpentinization). Cr reaches the highest concentration (4180 ppm) in MS and decreases to 880 ppm in PSP. On the other hand, Ni has the highest concentration in PSP (3900 ppm) and lowest in MS (1050 ppm). Co content is very homogeneous in PSP whereas shows a wider variation range in FS and, particularly, in MS where it reaches the highest concentrations (330 ppm). Cu, with a variation range between 2 to 75 ppm, has a similar behavior to Co. V content is very similar in the three rock groups (varying from 10 to 60 ppm) with the lowest concentrations occurring systematically in FS, whereas zinc content appears to be randomly distributed, although in a narrow range of variation (30-60 ppm).

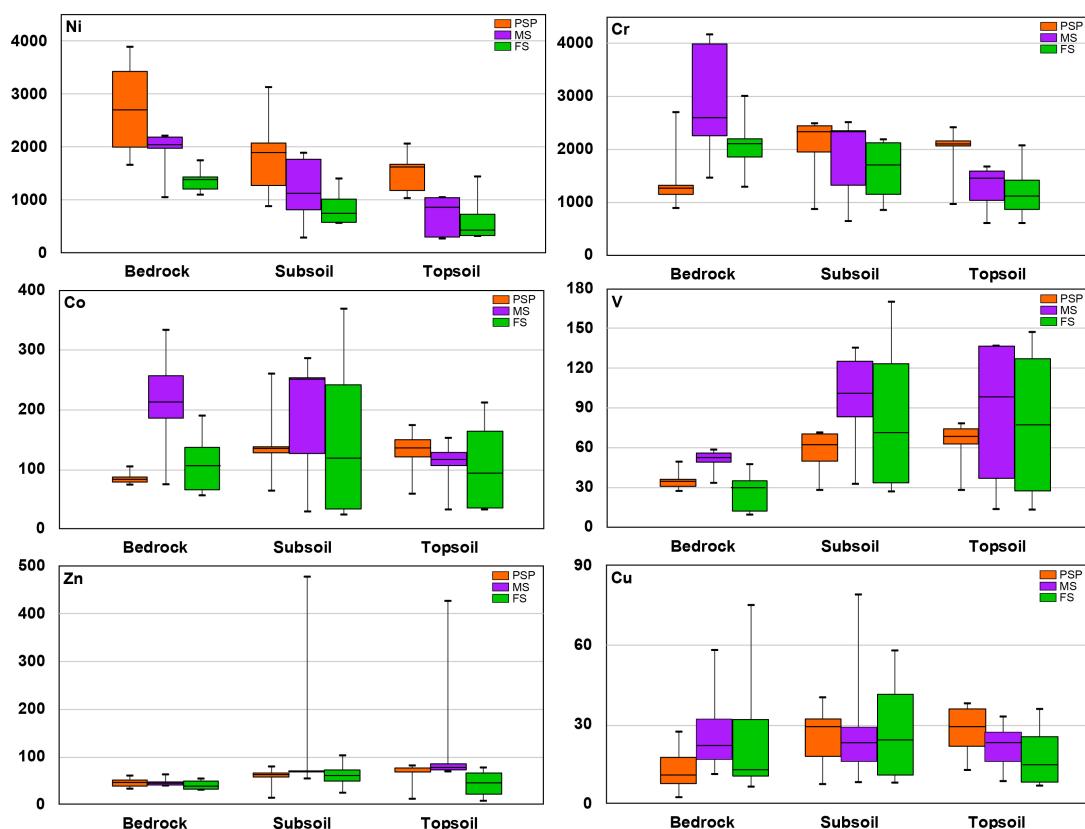


Fig. 3 - Box plots showing statistical parameters of concentrations (in ppm) of Ni, Cr, Co, V, Zn, and Cu for the studied soil profiles. Vertical lines show the range in concentrations, the boxes are bounded by the 1st and 3rd quartile, and the horizontal line inside the box represents the median value. Statistical parameters are based on 30 analyses. From Marescotti *et al.*, 2019.

These data highlight that the PTEs variability is primarily related to the petrologic and tectonic evolution but, at a local scale, also the mineralogical, lithological, structural, and textural features correlated to the degree of serpentinization and/or deformation significantly influence their distribution and concentration as also outlined by various authors (*e.g.*, Gulaçar and Delaloye, 1975; Shervais *et al.*, 2005).

PTEs in the ultramafic soil profiles

The studied soils soil profiles are weakly developed and characterized, from the bedrock toward the topsoil, by a thick saprolitic zone (up to 50 cm) with a gradual transition to the C horizon. The C horizon grade progressively toward the A horizon that is generally less developed. The O horizon, when present, is very thin (up to 5-10 cm; Fig. 4). The profile thickness varies from 40-60 cm in PSP, to 50-70 cm in MS and to 80-100 cm in FS. Soils developed on serpentinite bedrocks (MS and FS) were classified as Magnesic Leptic Skeletic Cambisols, whereas those developed on peridotites as Chromic Magnesic Leptic Skeletic Cambisols (FAO-WRB classification; IUSS, 2015).

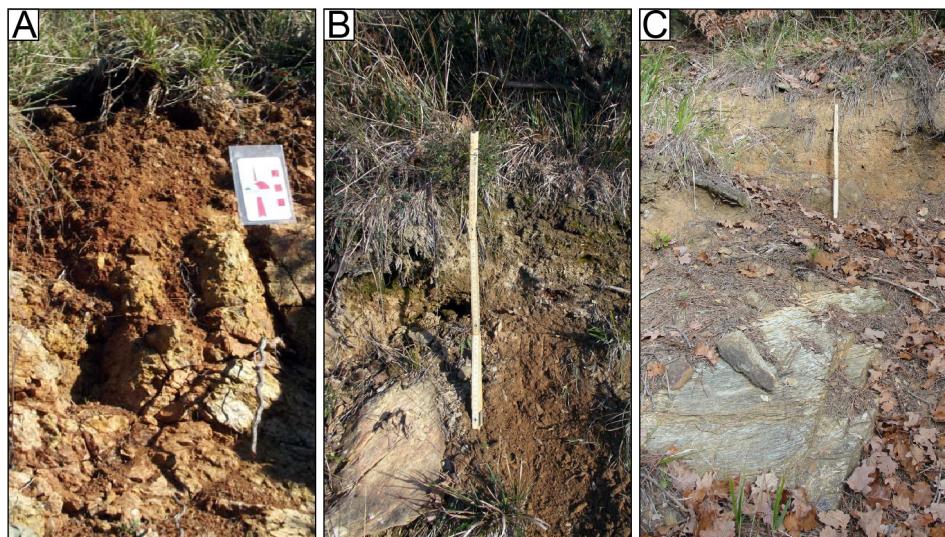


Fig. 4 - Studied ultramafic soil profiles developed on different bedrocks: A) PSP; B) MS; C) FS.

All the soils display heterogeneous granulometry, ranging from gravelly mud, muddy gravel, to sandy muddy gravel (Folk, 1954) with no clear relationships to the different bedrock types. The clay fraction is always a subordinate component both in topsoil and subsoil samples varying from 2 to 7 wt.%. The organic matter varies from 29% to 38% and is mostly present in the first 5 cm of the soil profiles. Conversely, soil color is strongly influenced by the bedrock nature. PSP soils vary in color from strong brown (7.5 YR 4/6) to reddish brown (10 YR 5/4). FS soils cover the widest range of the color varying from light grey (5 Y 7/2) to brown (7.5 YR 4/6), MS soil ranges in color from yellowish brown (10 YR 5/4) to dark brown (10 YR 3/3). These variations are strictly related to the weathering intensity and to the abundance of iron oxides.

Micromorphological analyses allowed to recognize 5 main group of clasts occurring within the soil skeleton (Fig. 5): *i*) lithorelics, *ii*) alterorelics, *iii*) alteromorphs, *iv*) iron crusts and nodules, and *v*) allochthonous clasts.

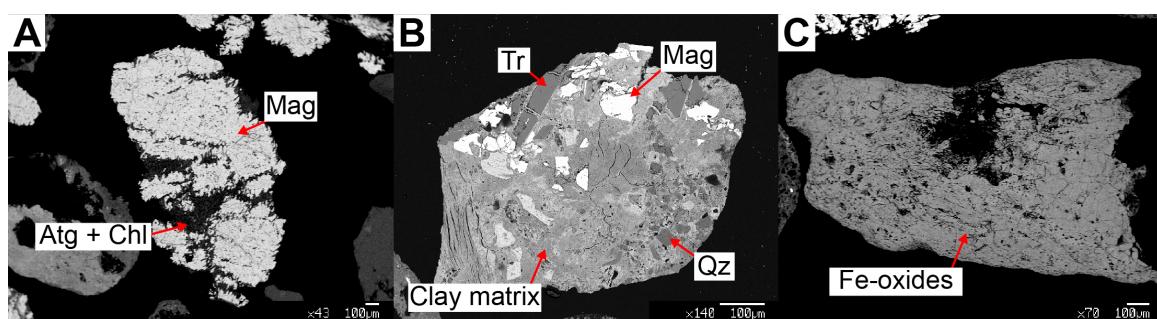


Fig. 5 Example of micromorphological features (SEM-BSE images): A) Alterorelics aggregate of magnetite and mixture of antigorite and chlorite, slightly altered and replaced by clay minerals and oxyhydroxides in topsoil of FS soil profile; B) Alteromorph composed by magnetite, tremolite, and quartz with clay-rich matrix in subsoil of FS soil profile; C) Iron crust composed by cryptocrystalline Fe-oxyhydroxides.

The soil profiles developed on the three different bedrocks are characterized by significant mineralogical variations both for the main primary and authigenic mineral species and for their relative abundances. Conversely, the mineralogical variations between topsoils and subsoils are less important and mainly concern the relative abundance between primary minerals inherited from the parent rocks and the new forming authigenic minerals. In general, the mineralogy of the skeleton is closely related to bedrock mineralogy as expected for primitive A-C soils). The clay fraction is mainly composed by amorphous and/or low crystalline phases, with subordinate clay minerals (mainly low crystallinity chlorite and subordinate illite) and goethite.

The studied PTEs are hosted both in primary and authigenic minerals. Among primary minerals, spinel-group minerals, antigorite, and chlorite represent the most important PTEs bearing primary minerals, whereas low crystalline to amorphous Fe-oxyhydroxides and clay minerals are the main PTEs-bearing authigenic phases.

Cr is mainly contained within spinel-group minerals (up to 378900 ppm), antigorite (up to 2250 ppm), and chlorite (up to 3530 ppm). In the authigenic phases, the highest and more variable concentrations were recorded in low crystalline to amorphous Fe-oxyhydroxides occurring as alteromorphs after spinel-group minerals. In the clay minerals, the variations in Cr contents are mostly related to the different mineral species with the highest concentrations (up 4750 ppm) in low-crystalline chlorites and the lowest (about 80 ppm) in illite

The principal Ni-bearing primary minerals in all soil types are spinel-group minerals (Cr-magnetite up to 3800 ppm, ferrian chromite up to 2320 ppm, and magnetite up to 3990 ppm), antigorite (up to 2610 ppm), and chlorite (up to 2850 ppm). The concentration of Ni in authigenic minerals is much more variable depending both on soil type and on the mode of occurrence. The highest and more variable concentration was recorded in Fe-oxyhydroxides, mostly represented by amorphous phases and crystalline goethite. The highest median concentrations were found in PSP soils (4800 ppm), up to one order of magnitude higher than in MS and FS soils. In MS and FS soils the highest Ni concentration (3840 ppm and 3700, respectively) was recorded in Fe-oxyhydroxides occurring within alteromorphs after magnetite and serpentinite clasts as well as in alterorelics, whereas the lowest concentration generally occurs in iron nodules and iron crusts. In the clay minerals, the variations in Ni contents are mostly related to the different mineral species with the highest concentrations (up to 3300 ppm) in low crystalline chlorites and the lowest (about 50 ppm) in illite.

Among the primary minerals, spinel-group minerals are also the most important Co-bearing phases in all the three soil types with median values ranging from 1210 ppm (PSP soils) to 530 ppm (MS soils). Antigorite and chlorite have relatively low Co concentrations; the highest median values (360 ppm) were recorded in antigorite occurring in pseudomorphic textures from PSP soils and in chloritized pyroxenes from MS and FS soils. Also, in the authigenic minerals Co concentrations are generally much lower than Cr and Ni with a similar behaviour both in clay minerals and Fe-oxyhydroxides.

As expected, the other PTEs (V, Cu, and Zn) are generally present in primary minerals in lower concentrations than Cr, Ni, and Co. The only exceptions are represented by the spinel-group minerals, which are characterized by significantly high median concentrations of V (up to 1810 ppm in PSP soils) and Zn (up to 3680 ppm in PSP soils). As for Cr, Ni, and Co, the authigenic minerals, both clay minerals and Fe-oxyhydroxides, resulted to be able to scavenge non-negligible amounts of PTEs. The highest median concentrations of V (1080 ppm), Cu (450 ppm), and Zn (3530 ppm) always occur in Fe-oxyhydroxides alteromorphs and alterorelics from PSP soils.

The presence and distribution of PTEs within the studied soils are typical of the ultramafic soils worldwide with very high concentrations of Cr, Ni, and Co, and subordinate but environmentally significant concentration of V, Cu, and Zn. In general, Cr and Ni contents decrease according to the serpentinization degree of the relative bedrocks, whereas Co, Zn, and Cu do not show clear correlations. Along the soil profile, Cr and Ni systematic decrease in concentration from subsoil (2010 and 1850 ppm respectively) toward topsoil (1220 and 640 ppm respectively) whereas Co distribution is more homogeneous

In summary, the high PTEs concentrations are linked both to the primary minerals, inherited by bedrocks, and to the stable authigenic products (Fe-oxyhydroxides and clay minerals). The different behavior of PTEs

along the studied soil profiles, is due both to the different weatherability of the primary minerals and to the relative mobility of the different metals involved in the pedogenetic processes. The authigenic minerals can play an important role in controlling their mobility. Considering the high stability of goethite, hematite and clay minerals in supergenic environment, it is evident that this mineral species are effective and often permanent traps for the most important PTEs occurring in ultramafic soils and bedrocks, thus reducing their bioavailability.

CONCLUSIONS AND GENERAL REMARKS

The results obtained in this work evidence that the concentration of Cr, Ni, and subordinately Co always exceed (up to one order of magnitude) the residential and industrial threshold values (CSC) according to Italian law (D.M. 471/1999; D.Lgs 152/2006; Fig. 6). Other detected elements of environmental concern (V, Cu, and Zn) are generally below these limits. However, the PTEs concentrations observed in the studied ultramafic soil profiles are to be considered of geogenic origin and are linked both to the primary minerals, inherited by bedrocks, and to their stable authigenic products (Fe-oxyhydroxides and clay minerals).

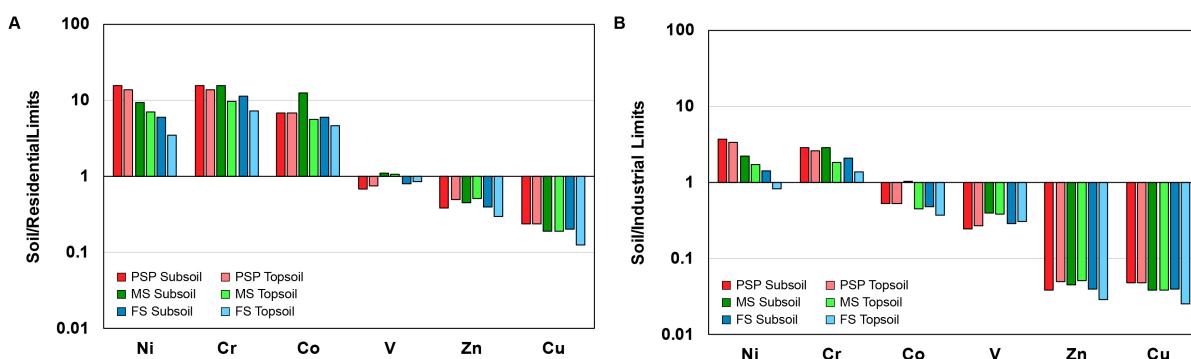


Fig. 6 - PTEs concentration normalized to Italian threshold values for residential (A) and industrial (B) sites according to Italian law (D.M. 471/1999; D.Lgs 152/2006). From Marescotti *et al.*, 2019.

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