Quantification of heavy metals in agricultural soils: the influence of sieving in standard analytical methods

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CONTEXT

Currently the productivity of some European cropping systems is maintained artificially by increasing production factors like mineral fertilizers or pesticides, using heavy machinery highly energy consuming and improving the technologies in order to mask the loss of productivity resulting from soil quality degradation. Conservation of agriculture soils is a topic of major concern, namely through the increase of soil organic matter. The SoilCare project (https://www.soilcare-project.eu/) aims to enhance the quality of agricultural soils in Europe, through the implementation and testing of Soil Improving Cropping Systems in 16 study sites. In Portugal, the application of urban sewage sludge amendments in agriculture soils has been investigated.

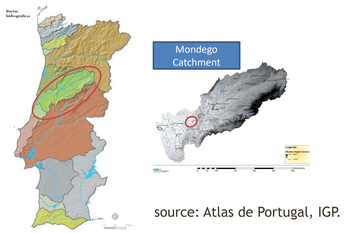
PROBLEM

However, the use of urban sludge as fertilizer in Agriculture is a sensitive topic, due to the risk of long term accumulation of heavy metals and consequent contamination of the soil. The recent Portuguese legislation (Decret-Law 103/2015) is more restrictive than the precedent one (Decret-Law 276/2009) in terms of maximum concentrations of heavy metals in agricultural soils for sludge application. The analytical quantification of heavy metals, however, raises some methodological questions associated with soil sample pre-treatment, due to some imprecisions in standard analytical methods. For example, the ISO 11466 regarding the extraction in Aqua Regia provides two pre-treatment options: (i) sieve the soil sample with a 2 mm mesh (but if mass for analyses is <2g, mill and sieve the sample <250µm is required), or (ii) mill and sieve the soil sample through a 150µm mesh. On the other hand, the EN 13650 requests soil samples to be sieved at 500µm. Since heavy metals in the soil are usually associated with finer particles, the mesh size used during the pre-treatment of soil samples may affect their quantification. This study aims to assess the impact of soil particle size on total heavy metal concentrations in the soil. .

METHODOLOGY

Soil samples were collected at 0-30cm depth in an agricultural field with sandy loam texture, fertilized with urban sludge amendment for 3 years. These samples were then divided in four subsamples and sieved with 2mm, 500µm, 250µm and 106µm meshes (soil aggregates were broken softly but soil wasn't milled). Finer and coarser fractions were weighted and analyzed separately. Heavy metals were extracted with Aqua Regia method, using a mass for analyze of 3g, and quantified by atomic absorption spectrophotometer with graphite furnace (Cd) and flame (Cu, Ni, Pb, Zn and Cr)

STUDY SITE

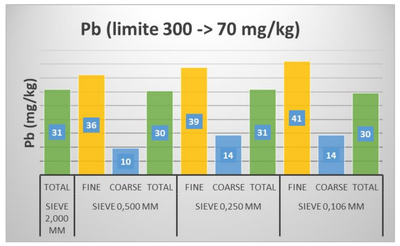


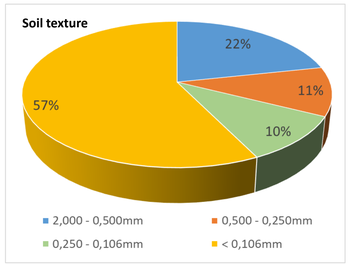
source: Atlas de Portugal, IGP.

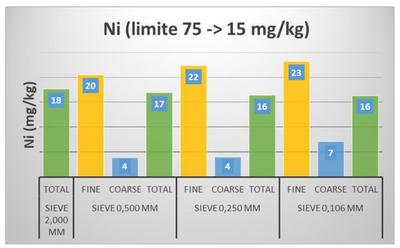
Study areas where were taken the soil samples for heavy metal analyses are located in the Mondego lower valley, an alluvium plane situated in Central Portugal. Soils are modern alluvial soils, with a texture from silt-loam to sandy-clay-loam. Climate is Mediterranean. The annual average temperature is 16.1ºC, and the annual average precipitation is 922 mm, with the particularity of intense rainfall events in autumn, that increase the lixiviation risk of contaminant to the groundwater.

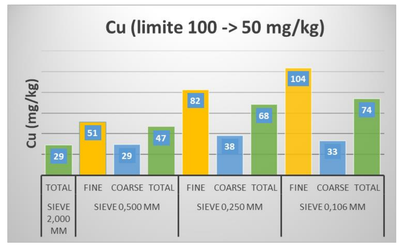
RESULTS

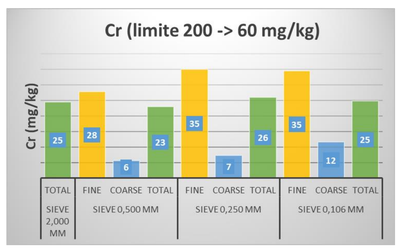
- Heavy metals concentrations increase linearly with the decline of the coarser fraction (Except for Cu).
  - Analyzing heavy metals content only in the finest fractions of the soil leads to an over estimation of their concentrations in the total soil.
- The coarser fractions of soil comprise low, but not negligible, concentrations of heavy metals.
  - Calculating heavy metal concentrations in the soil based on the weighted average of both fine and coarse fractions and associated concentrations, provide similar results to those driven by the analyses of heavy metals in the <2mm fraction.
- Sieving and analyzing only the finer fractions of the soil overestimate the quantification of heavy metals in total soil and for Ni, Cu and Zn considering the soil fraction <0,106mm only lead to exceed erroneously the maximum limits of the law.
  - Clearer indications on analytical procedures should be provided in analytical standards, in order to properly assess heavy metal concentrations and compare the results with soil quality standards legislated.

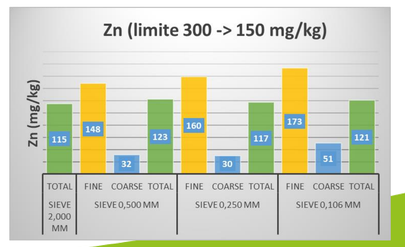












LIMITE mg/kg (Decret-Law 103/2015) -> (Decret-Law 276/2009)

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