



Linking bioavailability and toxicity changes of complex chemicals mixture to support decision making for remediation endpoint of contaminated soils

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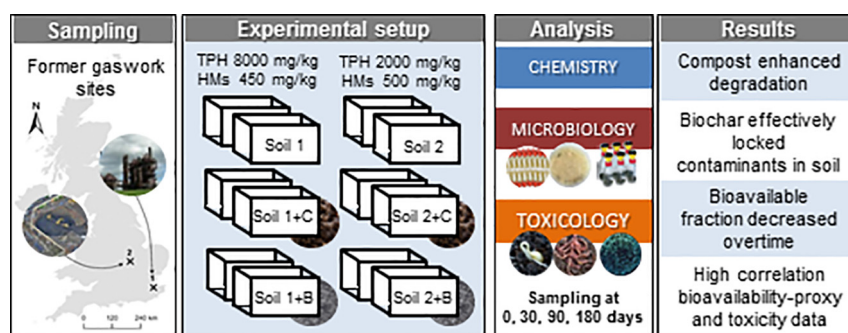
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HIGHLIGHTS

- Bioavailable fraction decreased over time in particular for 3–4 rings PAHs.
- Compost was found to be successful in enhancing degradation and reducing toxicity.
- Biochar was able to effectively lock organic contaminants in soil.
- Multiple lines of evidence needed to link bioavailability to end-point remediation
- High correlation between bioavailability-proxy and toxicity data

GRAPHICAL ABSTRACT



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ABSTRACT

A six-month laboratory scale study was carried out to investigate the effect of biochar and compost amendments on complex chemical mixtures of tar, heavy metals and metalloids in two genuine contaminated soils. An integrated approach, where organic and inorganic contaminants bioavailability and distribution changes, along with a range of microbiological indicators and ecotoxicological bioassays, was used to provide multiple lines of evidence to support the risk characterisation and assess the remediation end-point. Both compost and biochar amendment ($p = 0.005$) as well as incubation time ($p = 0.001$) significantly affected the total and bioavailable concentrations of the total petroleum hydrocarbons (TPH) in the two soils. Specifically, TPH concentration decreased by 46% and 30% in Soil 1 and Soil 2 amended with compost. These decreases were accompanied by a reduction of 78% (Soil 1) and 6% (Soil 2) of the bioavailable hydrocarbons and the most significant decrease was observed for the medium to long chain aliphatic compounds (EC_{16-35}) and medium molecular weight aromatic compounds (EC_{16-21}). Compost amendment enhanced the degradation of both the aliphatic and aromatic fractions in the two soils, while biochar contributed to lock the hydrocarbons in the contaminated soils. Neither compost nor biochar affected the distribution and behaviour of the heavy metals (HM) and metalloids in the different soil phases, suggesting that the co-presence of heavy metals and metalloids posed a low risk. Strong negative correlations were observed between the bioavailable hydrocarbon fractions and the ecotoxicological assays suggesting that when bioavailable concentrations decreased, the toxicity also decreased. This study showed that adopting a combined diagnostic approach can significantly help to identify optimal remediation strategies and

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contribute to change the over-conservative nature of the current risk assessments thus reducing the costs associated with remediation endpoint.

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1. Introduction

Anthropogenic activities are the main cause of release of potentially toxic compounds in soil, among which heavy metals, metalloids and petroleum hydrocarbon products are the ones mostly found at contaminated sites (Hou and Al-tabbaa, 2014). Remediation approaches at contaminated sites are promising strategies to mitigate the risks posed by the pollutants; in particular bio-stimulation and bio-addition are common practices aiming at improving soil quality; adding organic matter, delivering nutrients, balancing pH, and increasing water holding capacity, thus enhancing the degradation process (Y. Wang et al., 2017). Commonly used amendment for soil remediation include composted agricultural by-products and coal combustion products (e.g. biochars) (U.S. Environmental Protection Agency, 2007), which have been widely studied due to their sustainable, efficient, and cost-effective approach (Ahmad et al., 2014). Compost amendment is a common remediation strategy where organic materials, such as manure or decomposed organic matter, are added to contaminated soil to stimulate soil microorganisms and thus promoting transformation of hydrocarbons into less toxic compounds (Davie-Martin et al., 2017). Apart from providing a carbon source for the existing pool of bacteria, compost addition can also introduce new microorganisms presenting different catabolic activities that could potentially enhance the remediation of polluted soils (Baldantoni et al., 2017). Another common amendment strategy, is adding biochar, a carbon-rich material obtained from the decomposition of biomass in absence (or low exposure) of oxygen (Liu et al., 2015). Due to its highly porous structure and alkaline nature, biochar is able to immobilise soil contaminants, hence its frequent usage in soil remediation (Egene et al., 2018). When added to the soil, biochar causes the release of carbonates, phosphates, and hydroxyl ions because of its alkaline pH value of 7–10, thus favouring metal stabilisation. Both electrostatic (surface adsorption) and non-electrostatic (functional groups complexation) interactions are responsible for a decrease in metals mobility and bioavailability (Van Poucke et al., 2018).

While organic amendment have been shown to effectively improve degradation of pollutants in soil, remediation success has often been defined by reduction of total contaminant concentration (Kuppusamy et al., 2017) rather than bioavailable concentrations. However, the extent to which contaminants are bioavailable has significant implications for the clean-up targets and risk assessment, as receptors respond to the fraction of contaminant that is bioavailable rather than the total fraction (National Research Council, 2003). Bioavailability, the freely available fraction of contaminants in soil (Semple et al., 2003), is nowadays regarded as an important feature to integrate in risk assessment as it can help to explain contaminants partitioning and degradation in the environment (Ortega-Calvo et al., 2015). In this regard, soil bioassays have been largely used to determine the ecological effects of complex chemicals or mixtures in environmental samples, since they provide a rapid characterisation of the contaminants' bioavailable fraction (Mazzeo et al., 2014).

There is a need to find a pragmatic and practical integrated approach where biological and chemical measures of bioavailability are correlated rather than developed independently, supporting the necessity of several lines of evidence for robust and informed risk assessment (British Standards Institution, 2017). Ecotoxicological methods along with bioavailability have the potential to offer a cost-saving approach to contaminated land by applying relatively cheap bioassays to evaluate the potential effects of contaminants of concern, and to demonstrate that a contaminated site may not require further actions (Kim et al., 2014; Sarsby and Meggyes, 2009; Udovic et al., 2013). However, the majority

of these approaches focus often on single contaminant rather than mixtures. Thus, assessing and implementing bioavailability of complex chemical mixtures in order to reduce conservativisms of the traditional chemical-based approach, remains a challenge (Cachada et al., 2016; Kienzler et al., 2016).

Therefore in the present study, we investigated the effect of soil amendments (compost or biochar) on the behaviour and bioavailability of a complex tar mixture containing aromatics, aliphatics, heavy metals, and metalloids. This work aim at assessing the relevance of a different range of biological indicators to understand the implications for risk assessment and identifying the end-point remediation. Moreover, bioavailability-proxy and the toxicity data were further correlated to provide the necessary evidence that these tools may be suitable for predicting site-specific bioavailability of complex chemical mixtures.

2. Materials and methods

2.1. Sample collection and physico-chemical characterisation

Two soils were collected from two UK brownfield sites occupied by former gasworks in Kent (51° 18' 39" N 0° 43' 17" E, Soil 1) and Northamptonshire ((52° 20' 23" N 0° 39' 17" W, Soil 2). Prior analysis, soil samples were homogenised through 2 mm- sieve to separate large particles such as plant parts (roots, stems, and leaves), cobbles and pebbles. Each soil samples was divided and processed for analysis, individual air-dried samples were used for: Soil pH analysis (10 mL), particle size distribution (10 mL), loss of ignition (5 g), Total Nitrogen (TN) and Total Carbon (TC) (0.001 mg), Total Phosphorous (TP) and available phosphorous (AP) (5 g). Additionally, fresh soil samples were used for dry matter and water content analysis (5 g).

TN and TC in soil material were determined by combustion at approximately 900 °C in the presence of oxygen; the amount of nitrogen and carbon was then measured by a thermal conductivity detector (TCD) (BS EN 13654-2, 2001) using vario EL III Element Analyzer (elementar Analysensysteme GmbH, DE). Total phosphorous was measured with a hydrochloric/nitric acid mixture extraction and the phosphorus content was determined by a spectrometric measurement in solution (ISO 11047, 1998) with a Spectronic Helios Gamma (Thermo Electron Corporation, UK). Available phosphorous was measured by treating the soil with a 0.5 mol/L sodium hydrogen carbonate solution at pH 8.5, the extract was then analysed by spectrometry (ISO 11263, 1994). Ammonium-N, nitrate-N and were extracted from soil using a 2 mol/L solution of potassium chloride, the extract was analysed by spectrometry (Jackson et al., 1986).

Dry matter and water content in soil samples were measured by drying at 105 °C ± 5 °C for 24 h. The difference in mass of an amount of soil before and after the drying procedure was used to calculate the dry matter and water contents on a mass basis (ISO 11465, 1993). Soil pH was measured (Jenway 3540 pH/Conductivity Meter, Keison Products, UK) with 1 part of soil per 5 parts of water after shaking for 60 min and subsequent equilibration period of 30 min (ISO 10390, 2005). The soil organic content was determined by loss of ignition (LOI): air-dried soil was dehydrated at 105 °C for 24 h then ashed at 450 °C for 5 h and ignition loss was expressed as a percentage of the dehydrated sample (BS EN 13039, 2000). Particle size distribution was determined by the sieving and sedimentation method. In short, soil organic matter was decomposed with hydrogen peroxide and the resulting slurry dispersed with a buffered sodium hexametaphosphate solution, then the different particle size fractions were determined by a combination of sieving and sedimentation (ISO 11277, 2009). The corresponding soil texture

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