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Rapid assessment of regional soil arsenic pollution risk via diffuse reflectance spectroscopy

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ABSTRACT

Soil arsenic (As) contamination by anthropogenic and industrial activities is a problem of global concern. This pilot study demonstrates the feasibility of adapting the diffuse reflectance spectroscopy (DRS) approach using the visible near infrared (VisNIR) spectra for detecting soil As pollution. Further, spatial variability of soil As contamination was evaluated combining DRS based predictions and two geostatistical algorithms. The raw reflectance spectra were preprocessed using three spectral transformations for predicting soil As contamination using three multivariate algorithms. Quantitatively, better accuracy was produced by the elastic net-first derivative model ($R^2 = 0.97$, residual prediction deviation = 6.32, RPIQ = 7.33, RMSE = 0.24 mg kg^{-1}). The prediction of soil As was dependent on the close association between soil As and spectrally active soil organic matter and Fe-/Al-oxides. Moreover, the As pollution risks hotspots were reasonably identified using ordinary kriging and indicator kriging interpolations based on DRS predicted As values.

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1. Introduction

The lower Indo-Gangetic plain of India is historically known for its geogenic arsenic (As) problem (Amini et al., 2008). This As causes groundwater contamination which leads to environmental pollution as well as severe human health hazards (Kramar et al., 2015). Further, the landfill areas surrounding the major cities of this region like Kolkata are contaminated by anthropogenic As from sources like smelting factories, glass industries, and agricultural chemicals factories. In India, Krishna and Govil (2004, 2007) reported the magnitude and distribution of heavy metals in different industrial areas. Besides, Gowd et al. (2010) noted that uncontrollable dumping of waste followed by diffusion via rainfall and wind are mainly responsible for soil heavy metal pollution across vast urban areas of India. Sharma et al. (2007) documented the impact of wastewater use in Indian agriculture on soil heavy metal pollution. In the last decade, soil As contamination has been a severe environmental hazard in several countries of the world, especially in Bangladesh and India, affecting tens of millions of people (Smedley and Kinniburgh, 2002). Rock weathering along with atmospheric deposition has been identified as the major contributors of soil As pollution (Patel et al., 2005). Due to its widespread occurrence and high toxicity, there is a pressing need for rapid appraisal of soil As contamination for effective contingency planning (Mandal and Suzuki, 2002).

In contaminated soil, toxic metalloids like As often exist with other minerals and their oxides, microbes, and soil humic fractions. Previously, environmental soil scientists collected As contaminated soil samples in the field and analyzed them in the laboratory with complex, multi-step wet-chemistry methods via atomic absorption spectroscopy or inductively coupled plasma spectroscopy requiring caustic acid digestion. Despite their accuracy, these traditional methods are laborious, costly, time intensive, require both consumables and skilled analysts. Furthermore, they are impractical in many instances due to limited access to instrumentation.

Given the requirement for rapid and all-inclusive assessment of soil properties for natural resource management, techniques such as diffuse

Abbreviations: DRS, diffuse reflectance spectroscopy; RPD, residual prediction deviation; VisNIR, visible near infrared; PLS, partial least squares; PCR, principle component regression; RF, random forest; MSW, municipal solid waste; LOI, loss on ignition; LASSO, least absolute shrinkage and selection operator; ENET, elastic net; RMSE, root mean squared error; RPIQ, ratio of performance to inter-quartile distance; 1D, first derivative; 2D, second derivative; OSC, orthogonal signal correction.

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reflectance spectroscopy (DRS) using visible near infrared (VisNIR, 350–2500 nm) spectra are being tested as probable surrogates to substitute traditional soil testing protocols (Chang et al., 2001). Explicitly, VisNIR DRS involves the use of reflected light in the 350- to 2500-nm range. Reflectance spectra are parsed into discreet intervals (e.g., 1–10 nm) to construct reflectance profiles, which are then statistically compared with other quantitative soil parameter data to construct a predictive model for that parameter. Spectral sensors based on this approach are field-portable, cost-effective with almost zero recurring expenditure, mostly non-invasive, support high-throughput, and have the potential to be operated both in proximal and remote sensing mode. VisNIR DRS has already simultaneously characterized several soil attributes from reflectance values (Vasques et al., 2009). Since spectrally active chromophores like Fe-/Al-oxides, secondary clay minerals, humic fractions and carbonates often absorb or bind metal ions, DRS using infrared spectra in conjugation with chemometrics have been used to predict As and other heavy metals (Wu et al., 2005; Reeves and Smith, 2009; Ren et al., 2009; Niazi et al., 2015; Wang et al., 2014; Chakraborty et al., 2015). While correlating DRS spectra with soil As, Song et al. (2011) explored the covariation between soil organic matter and As content. Essentially, regression based predictive modeling via advanced chemometric methods like partial least squares regression (PLS), random forest (RF), principle component regression (PCR), support vector regression, and artificial neural networks (ANN) have been used for the quantitative elucidation of soil spectra (Moros et al., 2009). However, inconsistent prediction accuracies and absorption features were reported from different heavy metal DRS studies (Kooistra et al., 2001; Siebielec et al., 2004; Schwartz et al., 2011).

Moreover, while DRS-based toxic metal quantification has shown considerable promise, proper evaluation of spatial dependence among DRS-predicted toxic metal contents has not received much attention yet. A map of soil contamination for a specific contaminated area can be developed owing to the spatial autocorrelation among soil heavy metal contents. Since DRS can be used to rapidly quantify soil heavy metal contents, an effective conjugation between DRS and geostatistical techniques can be exploited to promptly identify spatial correlation among soil heavy metal contents, much quicker than conventional wet-chemistry combined with geostatistical methods. Studies have already identified the potential of DRS in conjugation with geostatistical tools for producing faster spatial prediction of soil Cd and petroleum hydrocarbon (Chakraborty et al., 2012; Chen et al., 2015).

Competing technologies chiefly concern portable X-ray fluorescence (PXRF) spectrometry which has been successfully used to quantify multiple soil elements on-site (Weindorf et al., 2014). Notably, while PXRF calculates the metal concentrations from the emitted fluorescence intensities (Potts, 2008), DRS indirectly computes the metals from their association with soil organic matter (SOM), secondary minerals and Fe-/Al-oxides (Choe et al., 2009; Horta et al., 2015). However, PXRF detection of As can be cumbersome in some instances due to overlapping spectral peaks between As and Pb. Also, compared to PXRF, DRS offers shorter dwell time (scan acquisition time 5 s vs. ~60 s) and produces no ionizing radiation. As such, no special operational permits are required for the use of DRS. Nonetheless, several recent studies have extolled the virtues of combining DRS with PXRF elemental data (Aldabaa et al., 2015; Chakraborty et al., 2015; Wang et al., 2015).

Therefore, this investigation was designed to: i) assess the potential of rapidly predicting soil As via DRS using VisNIR spectra in conjunction with three diverse spectral pretreatments and three predictive algorithms; and ii) explore the viability of rapidly detecting soil As pollution hotspots of a landfill area exploiting DRS in conjugation with two geostatistical estimation algorithms. We hypothesize that the combination of DRS and geostatistical estimation algorithms can be used to rapidly map the spatial variability of soil As content.

2. Materials and methods

2.1. Study area and data collection

The area chosen for As prediction and mapping is called the Dhapa landfill region, located in the eastern peripheries of Kolkata city, India (Fig. 1). Soils in this area are anthropogenic in nature, highly disturbed and analogous to similar landfill soils found in the United States (e.g., Typic Udorthents) or Europe (e.g., Technosols). Municipal solid wastes (MSW) of Kolkata and its suburbs (229 km^2) have been uncontrollably dumped in a huge disposal site (~26 km^2) over the last 35 years at an average rate of 3000 tons day^{-1}. These anthropogenic activities have resulted in leachate buildup in soil and leachate runoff in the adjoining water bodies (SCS engineers, 2010). The vast section of land adjoining the core area (~810 ha) is presently used for vegetable farming and provides the city ~147 t of vegetables per month. The major crops are cauliflower, ridge gourd, and corn. A total of 200 georeferenced surface soil (0–15 cm) samples were collected from these arable lands. A random sampling scheme was planned in ArcGIS 10.1 (ESRI Inc., USA) and sampling points were transferred into a portable Garmin etrex global positioning system receiver (Garmin Ltd., Schaffhausen, Switzerland). Soils were collected with core sampler and sample contact with oxygen was avoided as much as possible for meaningful quantification of redox-sensitive As species for future studies (Keon et al., 2001). The collected soil cores were immediately placed in N_2-purged field portable Aldrich® AtmosBag glovebags (Sigma-Aldrich India) for transportation to the laboratory.

2.2. Soil chemical analyses

In this study, we used AR grade chemicals, procured from Sigma-Aldrich India, and all required solutions were prepared with MilliQ (18.2 M Ω) deionized water (Chakraborty et al., 2014). To protect the samples for future speciation study, in the laboratory soil preparation and extraction steps were performed inside a stainless steel glovebox purged with ultra-high-purity N_2 gas (from liquid N_2) (Keon et al., 2001). Bigger debris was screened and soil samples were dried at 40 °C and grounded to <200 μm prior to chemical analyses and spectral scanning (Niazi et al., 2015). We avoided in-situ spectral measurements due to the presence of artifacts.

Total As content was determined by digesting ~1 g soil at 180 °C inside a microwave oven (Titan MPS, PerkinElmer, USA) with a mixture containing 10 ml each of HCl ($\rho = 1.19$ g/ml), HNO_3 ($\rho = 1.42$ g/ml), $HClO_4$ ($\rho = 1.68$ g/ml) and HF ($\rho = 1.49$ g/ml), (Song et al., 2012) and subsequent quantification via hydride generation-atomic absorption spectrometer (PerkinElmer, USA). Additionally, Fe_2O_3 and Al_2O_3 were quantified by an Agilent 7500 inductively coupled plasma-mass spectrometer (Agilent Technologies, USA). Measured As contents (mg kg^{-1} of soil dry weight) values were included as target variables in the subsequent DRS based regression models. Soil pH and EC [1:1 soil/water (v/v)] were estimated following Soil Survey Staff (2004), using a Systronics 335 digital pH meter and Systronics 306 conductivity meter (Systronics India Limited, India), respectively. Loss on ignition (LOI) content was determined after 8 h of ashing of soil at 400 °C (Nelson and Sommers, 1996); a temperature which promotes thorough organic matter ashing while minimizing dehydroxylation of minerals within the soil. Subsequently, the LOI was corrected for dehydroxylation of inorganic constituents through regression analysis from which SOM was estimated.

2.3. Spectral scanning and spectral treatments

Spectral reflectance of air-dried and finely grounded (<200 μm) soil samples was measured proximally over the VisNIR region (350–2500 nm) by a portable PSR-3500® VisNIR spectroradiometer (Spectral Evolution, USA). The reflectance data were resampled to 10 nm output

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