

## Geochemical speciation, anthropogenic contamination, risk assessment and source identification of selected metals in freshwater sediments—A case study from Mangla Lake, Pakistan



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### ABSTRACT

Geochemical speciation of selected metals (Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sr and Zn) using modified BCR sequential extraction procedure was performed in the sediments collected from Mangla Lake, Pakistan. The extraction method was optimized using certified reference material (BCR-701) which showed significant recoveries (95–106%). Among the metals, Cd, Co, Ni, Pb and Sr exhibited relatively higher mobility and bioavailability, while Cu, Fe, Mn and Zn were found mainly in the residual fractions. Principal component analysis (PCA) revealed four groups of the metals in the sediments; I (Cd–Cr–Ni–Sr), II (Co–Pb), III (Fe–Zn), and IV (Mn–Cu); first two groups were mostly contributed by anthropogenic sources. The pollution indicators (individual contamination factor, enrichment factor and geoaccumulation index) revealed significant anthropogenic contamination of Cd, Co, Ni, Pb, Sr and Zn in the sediments. Risk assessment code (RAC) indicated medium risk for Co and Ni; high risk for Cd and Pb; and very high risk for Sr in the sediments. Cluster analysis (CA) and global contamination factor (GCF) were used to identify the pollution hotspots, which indicated more severe metal contamination at sites near to Mirpur city and other urban/semi-urban areas (S3–S5), especially for Cd, Co, Ni, Pb and Sr.

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### 1. Introduction

Investigations pertaining to the metals in sediments have enlarged in recent years; while previously sediments were only considered as pollutant reservoirs (Passos et al., 2010; Nguyen et al., 2009). A minor fraction (about 1%) of these pollutants remains dissolved in water whereas overwhelming concentrations (99%) are stored in sediments. Therefore, sediments are the major sinks and carriers for contaminants in aquatic environments as some pollutants may be recycled through biological and chemical processes within the water column (Nemati et al., 2011a; Vink, 2009; Bartoli et al., 2011). The metals pollution of sediments is of major concern because of their toxicity, persistence and bio-accumulative nature and may be transferred to the overlying water and thereby entering into the food chain (Nemati et al., 2011a; Díaz-de Alba et al., 2011). In the absence of anthropogenic intrusions, trace metals in sediments are mainly associated with silicates and primary minerals and therefore have limited mobility. However, the metals introduced by human activities show greater mobility and are associated

with other sediment phases, such as, carbonates, oxides, hydroxides and sulfides (Passos et al., 2010; Heltai et al., 2005). Major sources of metals in aquatic systems are the rock weathering and anthropogenic activities including industrial wastewater, drainage of land, atmospheric inputs, soil erosion, biological activities and urban wastes (Díaz-de Alba et al., 2011; Carman et al., 2007).

Estimation of the total metal concentrations in sediments is not suitable to determine their mobility (Tüzen, 2003). The bio-availability and prospective toxicity of the metals to the biota depends on their chemical forms (Ahlf et al., 2009; Arnason and Fletcher, 2003). Therefore, researchers are interested in determining associations of the metals with different geochemical phases (Rauret, 1998). Though sequential extraction procedure is complicated and lengthy, it provides adequate information related to the origin, occurrence, biological/physicochemical aspects, mobilization and transport of metals (Passos et al., 2010). It emulates mobilization and retention of the metals in the aquatic ecosystems by changes such as, pH, redox potential and degradation of organic matter (Tessier et al., 1979; Förstner, 1985). Metals bound in different geochemical fractions are extracted by various reagents such as, inert electrolytes, weak acids, reducing/oxidizing agents and strong mineral acids in a sequence of increasing their extraction strengths (Passos et al., 2010). Various procedures reported in the literature

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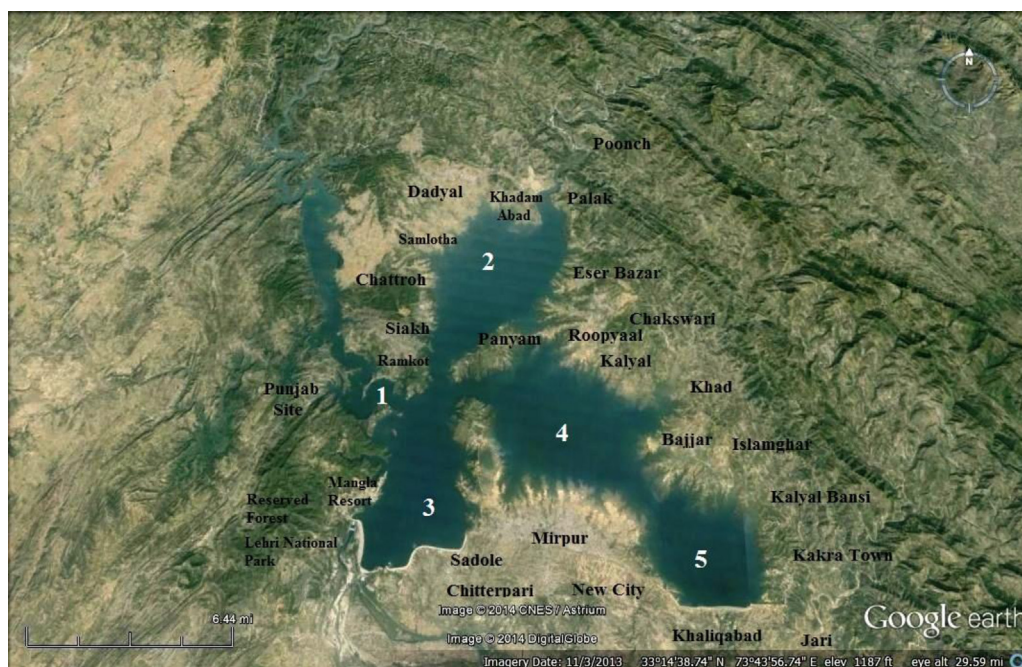


Fig. 1. Map of the study area showing sampling sites (1–5)

were standardized by European Community Bureau of Reference (BCR) in a multi-step modified sequential extraction procedure, which provides acid extractable, reducible, oxidisable and residual/immobile fractions of metals in the sediments (Razic and Dogo, 2010; Saracoglu et al., 2009; Ure et al., 1993). This method helps in indication about the mobility, biological availability and potential risks related to the metals contents in the aquatic ecosystems (Passos et al., 2010).

The main objective of the present study was to investigate the distributions of the selected metals (Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sr and Zn) in different geochemical fractions of the sediments from Mangla Lake, Pakistan, using the optimized three-stage mBCR extraction method. Different pollution indices (RAC, ICF, GCF, EF and  $I_{geo}$ ) were used in order to determine the contamination and risks associated with the metals in the sediments. Multivariate principal component and cluster analyses were also applied to the results in order to explore the associations between the metals/sites and their major contributing sources in the study area.

## 2. Material and methods

### 2.1. Sampling site

Mangla reservoir is one of the largest freshwater resources in Pakistan (Fig. 1). It was impounded in 1967 by damming the River Jhelum near Mirpur city, primarily for storing the water for irrigation and generation of hydroelectric power. Currently water is also used for drinking and household purposes in adjoining areas. The reservoir is fed by two perennial rivers (Jhelum and Poonch) and two non-perennial rivers (Kanshi and Khad). Reservoir being at the junction of these rivers has a square shape with its narrow flanks extending into the river valleys. The average water surface of the lake is approximately 26,500 ha and is divided into five sub-basins commonly referred as pockets, namely, Main Mangla, Jhelum, Poonch, Khad and Jari. The catchments areas of the reservoir are erosion prone resulting into high sedimentation rates in the reservoir thereby continuously reducing its storage capacity (Saleem et al., 2013).

### 2.2. Sample collection and storage

Surface sediment samples were collected from five major sites (S1 – 33°12' N, 73°38'E; S2 – 33°17' N, 73°42'E; S3 – 33°09' N, 73°40'E; S4 – 33°11' N, 73°45'E and S5 – 33°08' N, 73°50'E) of Mangla Lake, Pakistan in winter 2012. A total of 80 composite surface sediments samples (1–15 cm, top layer) were collected in pre-cleaned Ziploc polythene bags using a snapper ( $\Phi$  5 cm). Each composite sample was composed of 5–7 sub-samples collected within an area of 20–30 m<sup>2</sup>. The samples were kept in plastic ice-cold containers. Large objects including stones, pieces of brick, concrete and cinders were manually removed. The sediment samples were oven dried, grounded, homogenized, sieved through a 2 mm mesh and sealed in clean polythene bags and then stored in a refrigerator until further processing (Nemati et al., 2011a; Kannan et al., 2008). Every precaution was taken to avoid contamination during sampling, drying, grinding, sieving and storage.

### 2.3. Reagents and glassware

All reagents used were of analytical grade (certified purity >99.9%) procured from E-Merck, Germany or BDH, UK. Deionized water was used for the preparation of working standards at the time of analysis by successive dilution of the standard metal solutions (1000 mg/L). All laboratory glassware (Pyrex) was cleaned using a HNO<sub>3</sub> (20%, v/v) bath overnight, followed by repeated rinsing with doubly distilled water (Díaz-de Alba et al., 2011). For the removal of adherent organic impurities from glassware, they were rinsed with acetone. Finally the glassware was dried in an electric oven maintained at 80 °C for about eight hours prior to use.

### 2.4. Physicochemical characterization

Sediment samples were air-dried and sieved through a 2 mm sieve. Electrical conductivity (EC) and pH were measured with a multimeter (Bench Meter, Martini Instrument Mi 180) in a 1:2 sediment:water suspension (Radojevic and Bashkin, 1999). Organic matter content (OM) was estimated by measuring the loss of weight on ignition at 550 °C (Díaz-de Alba et al., 2011).

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