

# Determination of Heavy Metals in Saline Soil: A Comparison between Microwave Assisted Single Extraction and Sequential Extraction Method

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## Abstract

The present research assesses the chemical fractionation of cadmium (Cd), chromium (Cr), and lead (Pb) detected in saline soil samples of Hyderabad Pakistan. Based on the reagents used in the BCR three step sequential extraction scheme (BCR-SES) for partitioning Cd, Cr, and Pb in saline soil samples, a Microwave single step extraction (MSE) method was developed. The validity of the methods was confirmed using certified reference material (CRM) BCR-701 and the standard method of addition (recoveries 96-102%). The data provided by the BCR-SES method for Cd, Cr and Pb were compatible with MSE using the same operating conditions applied and checked in BCR fraction by the Certified Reference Material (CRM) BCR 701. Atomic absorption spectrometry was used to test extractable Heavy Metals (HMs) substances obtained by comparative method. Using compromised sonication and microwave conditions, steps 1–3 of the sequential extraction (excluding hydrogen peroxide digestion in step 3) could be accomplished with ultrasonic bath between 15–30 min, whereas 60–120 s were needed for MSE. The total concentrations of Cd, Cr, and Pb extractable metals produced by three separate extractions ranged from 1.02-3.30, 5.02-9.95 and 3.97-8.01  $\mu\text{g/g}$  respectively. The accuracy of the proposed BCR single extraction procedure (RSD %) for all metals was found within the % range from 4.91–8.61% respectively.

**Keywords:** BCR sequential extraction; Microwave single extraction; Heavy metals; Saline soil; Electrothermal atomic absorption spectrometry

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## Introduction

The beginning of the 21st century is characterized by a water shortage of world, contamination of the environment and rising salinization of soil and water. Increasing human population and reduction in land available for cultivation are two threats for agricultural sustainability [1]. High levels of heavy metals in soils, and through transfer processes, also in groundwater and plants, may have a negative effect on animals and human health [2]. The overall concentration of metals in contaminated environmental samples is a weak marker of their bioavailability, mobility or toxicity; such characteristics generally depend on the various chemical modes of linking between trace metals and solid sample phases. There is detailed analysis of various extraction methods of metals from environmental samples [3]. The quantity of

environmental research samples collected in routine monitoring or risk and mitigation assessment studies is therefore gradually growing. The first step is to assess whether the total concentration of metals is in the range of background levels, or in compliance with national legislation, above the concentration limits [4]. As the Environmental Protection Agency (EPA) has approved microwave assisted procedure with nitric acid [5].

In the soils the process of metal leaching was commonly used. Nitric acid is adequately strong to solubilize ash metals [6]. Sequential extractions offer semi-quantitative data on distributions between geochemical soil and waste products between operationally specified fractions. While sequential extractions are being used to deduce heavy metal speciation, sequential extraction fractions do not typically represent the actual speciation of chemical materials

[7]. Soil metal ions shall be divided among various phases such as organic matter, iron oxydohydroxides, aluminum (Al) and manganese (Mn) carbonate and sulphides. Additionally, metal ions are maintained by different mechanisms (ion exchange, adsorption, precipitation and co-precipitation) on these solid phases [8]. Partial extractions, unlike sequential extraction scheme, are an easy and cost efficient way to explore the labile metals in soil. While there are several partial extraction approaches, they usually fall into three classes: (i) dilute solutions of strong mineral acids, (ii) weak acids (iii) solutions of reducing agents [9].

The BCR method aims for metals fraction into acid extractable, reducible, oxidizable and residual, which are operationally specified, with steps to target metals that are exchangeable and carbonates, iron and manganese related oxide/hydroxide metals, sulfide-bonded metals, and organic materials, respectively, and minerals [10]. Many of them considered to be of major environmental significance as Cd, Cr and Pb were heavy metals found. Our initial step was to test the methods by adding a wide variety of CRMs covering a broad range of environmental matrices including salt soil. The second step was to extend the techniques to a collection of samples of saline soils [4]. The key drawbacks of sequential extraction methods are that they take a very long time and are rarely applied for regular analysis. This question was also noticed and substitutes the conventional procedure with other solutions, such as microwave heating (MW) [11]. Microwave energy is an incredibly useful source of auxiliary energy, commonly used for analysis of heavy metals. In recent years, methods of microwave extraction for the speciation analysis of elements in geological and ecological samples have been implemented to improve extraction methods and reduce the treatment duration [12]. Nevertheless, the lack of evidence for the stability and bioavailability of toxic metals in soil or water continues the application of microwaved extraction methods [13]. The main goal of current study was to compare the difference achieved by using BCR-SES, the use of identical operating conditions and time-saving extraction MW oven for samples of saline soil. BCR 701 Soil certified reference material was also used for accelerated procedures extraction performance. The Cd, Cr and Pb amount in extracts have been analyzed by electrothermal atomic absorption spectrometry (ETAAS).

## Experimental Design

### Reagents and glassware

The deionized water were used in the research center was taken by a purifier of Milli-Q (Millipore Corp, Bedford, MA United States). The following expository test reagents were taken: corrosive acid refrigerant (100%), corrosive hydrochloride (37%, specific gravity 1.4), corrosive nitric acid (65% sp. gr. 1.19), hydrogen peroxide (30%) and calcium chloride. Deutschland, Darmstadt. It was checked that metal defilements could be imagined. Sigma Aldrich Co obtained ammonium acetic acid derivation. Ltd. Limited. Weakening certified standards (1,000 ppm, Fluka Kamica) arrangements for the comparison of metal particles

have resulted in the arrangement of Cd, Cr and Pb. Recently, the expository review of hydroxyl ammonium chloride (Merck, Poole, UK) took place sometime before. We used 50 ml centrifugal polyethylene tubing (Bibby Sterilin Ltd, UK) for extractions, as well as a flat carafe and power shaker (Gallenkamp). WIROWKA Laboratoryyna sort WE-1 has been centrifuged to Mechanika Pheczyjna, Poland (speed run 0 6000 rpm, clock 0-60 minutes, 220/50HZ). To accelerate absorption a time-programmable home microwave oven (PEL PM023, 100 to 900 W Japan) was used. For pH change of the reagents and pH soil insurance, a WTW pH meter was used. The determination of Cd, Cr and Pb was carried out by electrothermal atomic absorption spectrometry (ETAAS) equipped with graphite furnace in digested soil samples. Hollow cathode lamps were used as radiation sources. The lamp power, band-pass width and other analytical parameters were set as suggested by the manufacturer (Table 1).

### Sample collection

The study areas situated in between 68.28°E – 68.32°E longitudes and 25.43°N – 25.44°N, latitudes along with the lower Indus Valley of Sindh. Most of the soil of this area is saline and is Indus Basin inherent, as it was developed during the process of soil formation. At the same two three samples of bulk soil (BS) and coded bulk soil (BS1), (BS2), (BS3) and (BS4). For the preparation of representative sample, three to four sub samples (1.0 kg each) were sampled from different sites of each location of saline soil. Soil samples were collected in the polypropylene zip bags. The collected each soil sample was spread on the clean plastic tray and dried in fume cabinets at ambient temperature. The dried soil samples were stored in cleaned polypropylene bags [14].

### Microwave Induced Single Extraction (MSE)

Long-standing successful use of microwave energy to improve sample preparation has been illustrated by the rise in sample temperature [15]. Microwave energy is an extremely useful source of auxiliary energy, commonly used by analytical chemists [16]. It was studied that a MW oven could be used during a single extraction stage of the BCR procedure. As required by the BCR method, these extractions are carried out at ambient temperature; the MW power optimization and time for extraction have been carried out with careful monitoring of the extraction solution temperature [17]. In all three individual steps in the BCR process, the control power of MW and time is retained, where the temperature is not above 50°C and the solutions have never been boiled. Via each complete process, blanks (reagent without samples) were also taken. The metal content of each single fraction was determined by subtraction of the results obtained in two consecutive steps in each proposed individual extraction method, with the exception of the 1st step (Exchangeable Metals) of the BCR procedure. For example, the amounts extracted metals with step 1 reagent were removed from those extracted with step 2 reagents in order to approximate the 'reducible fraction' of single extractions in the BCR method (0.5 M NH<sub>2</sub>OH·HCl). Metal recovery has in all cases been measured as the given ratio:

% Recovery = (Metal extracted by proposed single extraction / Metal extracted by Sequential BCR) x 100

Until extraction, 0.5 g of certified BCR 701 and saline soil samples were specifically weighted in the bottles and put in the oven ( $105 \pm 2^\circ\text{C}$ ) to a steady weight. This treatment resulted in a 2.5 percent weight loss in BCR 701 and various values for saline soil samples. Both subsequent calculations have been corrected to take those losses into account.

### Analytical figure of merit

The calibration curve for Cd, Cr and Pb were studied from their quantification limit to 1000  $\mu\text{g/L}$ . The regression relations of calibration for Cd, Cr and Pb were  $y = 16.9 \times 10^{-3} (\text{Cd}) + 10.3 \times 10^{-3}$ ,  $y = 7.5 \times 10^{-3} (\text{Cr}) + 7.5 \times 10^{-4}$  and  $y = 12.5 \times 10^{-3} (\text{Pb}) + 14.1 \times 10^{-4}$  respectively, and have quantitative coefficient of determination ( $R^2 > 0.99$ ). The limit of detection (LOD) and limit of quantification (LOQ) was calculated by the multiplication three and ten time with the signal to noise ratio, which is equal to  $s/m$  ( $s$  = standard deviation of ten blanks and  $m$  = slope of the calibration curve). The LOD/LOQ for Cd, Cr and Pb were found to be 0.324/1.02, 0.391/1.83 and 3.38/11.1  $\mu\text{g/L}$ , respectively.

## Results and Discussion

### Optimization of proposed procedures Microwave single extraction (MSE) procedure

The data collected from the extraction mixture (extractants – sample) in a MW oven were compared to those obtained by SES process at 20 – 80% of the power (900 W). It was observed that the maximal recovery of all HMs was observed in the first phase of the BCR protocol at 60 s heating period except when Pb attained high recovery after 90 s, longer recovery time up to 120 s, does not improve recovery of Cd, Cr and Pb. The highest recovery of Cd and Cr was obtained in reducible fractions at 90 s, while Pb required 120 s for favorable values. Cd and Cr recover at 70 s in the oxidizable portion, while Pb recovery at 90 s was obtained. Therefore, it was noticed that the Pb needed longer time for optimal matrix recovery [11]. Consequently, the most suitable conditions of extraction for all three MSE phases were identified for BCR 701 and bulk saline soil samples (BS) at 60–120 s and 60% of total 900W power. The power values of MW of the nominal 20–40 percent did not release HMs in quantitative amount. It was found that the sudden increase in temperature at 80 percent of the total power induces drastic changes in the pattern of metal extraction and fractionation, but while using 60 percent of the total power, the temperature did not shift significantly, so we choose 60 percent of the total power of the MW oven for all studies to release all HMs at maximum values relative to those obtained from the SES procedure [18].

### Comparison between BCR sequential extraction method (SES) and Microwave single extraction (MSE) method for extraction of Cd, Cr and Pb

After optimization of the MSE, the concentration ranges of Cd, Cr and Pb and their mean values were obtained. The data obtained in relation to the suggested MSE procedure could be compared with SES, extractable metal concentration in BCR 483. The data of HMs collected from SES were used to measure the percentage

of HMs recovered through all three single extraction techniques. The results of current study are reported as mean values ( $\mu\text{g/g} \pm \text{SD}$ ) in **Table 2**. There were no major deviations for  $p = 0.05$  when comparing the results shown by the proposed single extraction procedure and the conventional SES process (paired t-test). In general, the relative standard deviation (RSD %) values obtained via the MSE and SES procedures were comparable. The data obtained with the MSE have been compared to those of the SES for the soil and also for some of the CRMs used during the validation step. The purpose of the study was to assess the quality of the methods for the analysis, in relation to total testing time, operational problems and costs, taking account of the data collected.

### Acid soluble fraction

The acid soluble fraction is the 1st step of the SES procedure and is therefore usually extracted directly, data presented in **Table 2**. Thus, a mild acid was used to dissolve CO-23 without dissolving the aluminosilicates, Fe/Mn oxides and organic matter [19]. The MSE indicates the lower recovery of Cd and Pb (96.0% and 96.2%) while the Cr (101%) obtained higher recovery. The extraction efficiency is rise significantly for Cr due to the short extraction exposure time, which permits a less adsorption process in this stage [20].

### Reducible fraction

The reducible fraction reflects the metals that are bound to Fe and Mn oxides produced as the matrix undergoes reduced nature [19]. In contrast to SES values, the reducible fraction derived from the MSE showed variation in HMs values. The reducible fractions of MSE heavy metals indicate that the recoveries for all metals varied % from 98.5–102.

### Oxidizable fraction

The oxidizing fraction includes organic matter and the toxic metals found in sulfides that can be quickly released under oxidizing nature. The oxidizable fraction analyzed by MSE shows certain variations in the efficiency of the extraction of HMs extracted by MSE. Comparing the amount of HMs released by MSE with those acquired by the SES procedure ranged between 98.8% and 104%, the slightly higher concentrations of Cd (104%) and Pb (103%) were released from those obtained by the MSE method.

### Residual fraction

The non-extractable or residual metal fraction is contained within crystallized oxides and within crystal mineral lattices Cd, Cr and Pb have been observed to be 96.3–102% higher, respectively (**Table 2**).

### Total extractable heavy metals

Total extractable amounts of HMs derived from the SES method and those analyzed by MSE is shown in **Table 2**. According to the above it can be inferred that the MSE finds some major variations in the metal fractions [19]. In the case of MSE, all HM recoveries

**Table 1** Measurement conditions for electro-thermal atomic absorption spectrometer.

Parameters	Cd	Cr	Pb
Lamp Current (mA)	7.5	7.5	7.5
Wave length (nm)	228.8	357.9	283.3
Slit width (nm)	1.3	1.3	1.3
Cuvette	Cup	Cup	Cup
Dry	80-120/15	80-120/15	80-120/15
Ash	300-600/15	300-600/15	300-600/15
Atomization	1500-1800/5	1500-1800/5	2000-2100/5
Cleaning	1800-2000/2	1800-2000/2	2100-2400/2
Chemical modifier	Mg(NO <sub>3</sub> ) <sub>2</sub> + Pd(NO <sub>3</sub> ) <sub>2</sub>	Mg(NO <sub>3</sub> ) <sub>2</sub>	Mg(NO <sub>3</sub> ) <sub>2</sub>

**Table 2** Quality control of data using measured, certified, values for extractable amounts in certified reference material BCR-483 and comparative results (% recovery).

Steps/element	Values	Cd	Cr	Pb
F1	MSE values <sup>a</sup>	9.98 ± 0.04	10.1 ± 1.14	0.76 ± 0.11
	BCR-SES <sup>b</sup>	10.4 ± 0.16	9.96 ± 0.35	0.79 ± 0.09
	%	96	101	96.2
F2	MSE values <sup>a</sup>	24.9 ± 2.03	639 ± 0.45	385 ± 1.91
	BCR-SES <sup>b</sup>	24.3 ± 1.05	649 ± 0.46	381 ± 11.9
	%	102	98.5	101
F3	MSE values <sup>a</sup>	1.32 ± 0.07	1201 ± 12.4	72.4 ± 0.12
	BCR-SES <sup>b</sup>	1.27 ± 0.09	1215 ± 18.1	70.5 ± 0.18
	%	104	98.8	103
R	MSE values <sup>a</sup>	0.411 ± 0.05	191 ± 9.98	75.9 ± 0.21
	BCR-SES <sup>b</sup>	0.425 ± 0.04	188 ± 8.98	78.8 ± 4.51
	%	96.7	102	96.3
Σ Three steps + residual		36.6 ± 2.19	2041 ± 23.9	534 ± 2.35
Pseudo-total		37.2 ± 1.11	2051 ± 32.6	541 ± 5.59

**Table 3** Fractionation of toxic metals by proposed single extraction methods in Bulk soil (BS).

Samples	Metals (µg/g)	Parameters	Acid soluble	Reducible	Oxidizable	Residual	Σ three steps+ residual	Pseudo-total
BS1	Cd	Concentration	0.34 ± 0.07	0.87 ± 0.08	0.33 ± 0.09	0.38 ± 0.06	1.92 ± 0.27	1.97 ± 0.41
		Percentage (%)	17.7	45.3	17.2	19.8		
	Cr	Concentration	0.65 ± 0.06	2.17 ± 0.19	1.34 ± 0.04	0.90 ± 0.01	5.06 ± 0.17	5.02 ± 0.24
		Percentage (%)	12.9	42.9	26.5	17.8		
	Pb	Concentration	0.30 ± 0.03	1.23 ± 0.22	1.36 ± 0.14	1.10 ± 0.09	3.99 ± 0.51	3.97 ± 0.33
		Percentage (%)	7.51	30.8	34.1	27.6		
BS2	Cd	Concentration	0.19 ± 0.07	0.62 ± 0.03	0.20 ± 1.01	0.24 ± 0.07	1.25 ± 0.91	1.26 ± 0.22
		Percentage (%)	15.2	49.6	16.0	19.2		
	Cr	Concentration	1.09 ± 0.03	5.20 ± 0.02	1.93 ± 0.05	1.05 ± 0.12	9.27 ± 0.09	9.29 ± 0.33
		Percentage (%)	11.7	56.1	20.8	11.3		
	Pb	Concentration	0.59 ± 0.05	1.15 ± 0.04	1.48 ± 0.03	1.56 ± 0.03	4.78 ± 0.14	4.96 ± 0.36
		Percentage (%)	12.3	24.1	31	32.6		
BS3	Cd	Concentration	0.50 ± 0.18	1.39 ± 0.05	0.78 ± 0.09	0.71 ± 0.08	3.38 ± 0.40	3.30 ± 0.29
		Percentage (%)	14.8	41.1	23.1	21		
	Cr	Concentration	0.30 ± 0.05	3.96 ± 0.07	1.99 ± 0.03	1.81 ± 0.07	7.43 ± 0.23	7.42 ± 0.35
		Percentage (%)	4.03	53.3	26.8	24.4		
	Pb	Concentration	0.81 ± 0.06	1.70 ± 0.06	4.14 ± 0.04	1.19 ± 0.06	7.84 ± 0.18	8.01 ± 0.41
		Percentage (%)	10.3	21.7	52.8	15.2		

Samples	Metals ( $\mu\text{g/g}$ )	Parameters	Acid soluble	Reducible	Oxidizable	Residual	$\Sigma$ three steps+ residual	Pseudo-total
BS4	Cd	Concentration	$0.11 \pm 0.05$	$0.57 \pm 0.11$	$0.20 \pm 0.10$	$0.18 \pm 0.05$	$1.06 \pm 0.30$	$1.02 \pm 0.13$
		Percentage (%)	10.4	53.8	19.6	17		
	Cr	Concentration	$0.88 \pm 0.13$	$4.46 \pm 0.02$	$2.08 \pm 0.01$	$2.50 \pm 0.09$	$9.92 \pm 0.25$	$9.95 \pm 0.15$
		Percentage (%)	8.87	45	20.9	25.2		
	Pb	Concentration	$0.61 \pm 0.05$	$3.00 \pm 0.10$	$1.60 \pm 0.10$	$1.74 \pm 0.05$	$6.95 \pm 0.30$	$6.98 \pm 0.45$
		Percentage (%)	8.78	43.1	23	25		

range from 98.4% to 99.5% in decreasing order Cd > Pb > Cr than the values obtained by SES method.

### Application of single extraction procedure to saline soil

Optimizing of three steps of single and sequential extractions using adapted BCR procedures dependent on the use of a MW oven on saline soil samples was performed. Toxic metals (Cd, Cr and Pb) were extracted in BS1, BS2, BS3 and BS4 during the first stage within the range of (10.4-17.7, 4.03-12.9, 7.51-12.3%), respectively, in all the saline soil samples examined, describing the carbonate-bound metal (Table 3). According to results, a small portion of Pb was available as an acid-exchangeable material in all saline soil samples. The percentage of the metals in the extractable acetic acid fraction was in sequence Cd > Cr > Pb. In reducible step the main correlations of all HMs were noticed. The Fe and Mn oxides surfaces have a particular affinity to the natural pH of the cations. Fe and Mn hydroxides are effective soil scavengers for these metals [21]. In the case of oxidizable Cr and Pb fractions, the largest proportion was found compared with Cd. As per findings it can be assumed that Cr and Pb in all saline soil samples are the most mobilizable metals.

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## Conclusion

The main goal of sequential extraction methods offers valuable knowledge about the possible toxicity of heavy metals when working with saline soil. By eliminating sequential treatment with single extractions, the lengthy treatment needed in these techniques could be reduced. The use of single extractions and BCR sequential procedure yields similar outcomes to the conventional sequential protocol. At the point when the single extraction technique was utilized, the washing steps were likewise evacuated after each consecutive extraction stage which helped us to accelerate the testing methodology. The utilize of single extractions would subsequently permit the assessment of the extractable metals in saline soil tests and may well be valuable for a fast screening of the conceivable versatility and bioavailability of metals within the saline soil.

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