



An evaluation of the modified BCR sequential extraction procedure to assess the potential mobility of copper and zinc in MSW

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ABSTRACT

Copper and zinc were determined in municipal solid waste (MSW) samples with different deposit ages from *Tianziling* landfill site. The pseudototal metal contents of the MSW samples were determined following an aqua regia digestion. Operational speciation was performed using the modified BCR sequential extraction procedure. Analyses were carried out by AAS. Agreement between most of triplicate samples was acceptable. The amount of copper and zinc extracted in the sequential procedure (i.e. Step 1, Step 2, Step 3, residual) did not generally agree well with pseudototal digestion. Various MSW samples contained significant different levels of copper and zinc, but these were with different potential migrations. For example, 49.88%–76.34% of copper existed in five MSW samples was present as oxidizable fraction while ~40% of zinc was present as acid soluble fraction. The study illustrates the feasibility and importance of modified BCR sequential extraction procedure used as evaluation method when assessing the potential mobility of heavy metal in MSW landfill.

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1. Introduction

The problems of heavy metal leaching from municipal solid waste (MSW) landfill are of increasing concern in the world. It does not constitute a frequent groundwater pollution problem at landfill sites, partly because landfill leachate usually contains only modest concentrations of heavy metals and partly because the heavy metals are subjected to strong attenuation by sorption and precipitation mechanisms [1]. However, changing environmental conditions at the landfill site, i.e. leachate recirculation, can induce non-linear behavior and the sudden release of heavy metal that reach problematic levels [2], the so-called ‘chemical time bombs’ (CTB) [3]. Generally, only a small portion of heavy metal deposits in a landfill will have been leached and thus the MSW landfill will still have a large potential of contamination, even for hundreds to thousands of years [4]. However, heavy metal will be leached out entirely from the MSW landfill at some day in future eventually. Continued attention to heavy metal after deposition of MSW is necessary [5]. Therefore, it is important to assess the leaching behavior of heavy metal in MSW landfill.

Nowadays, the determination of total concentrations of metals has been established cannot give sufficient information to assess the environment impact of substrates studied [6–8]. The distribution of

heavy metals in the various phases determines their behavior in the environment: their mobility, bioavailability and toxicity [7,9,10]. Therefore, fractionation is also necessary to evaluate the leaching behavior and environmental risks of heavy metal in MSW landfill.

At present, the only laboratory routine available to determine metal binding forms is sequential extraction. It has been widely used for the fractionation of heavy metals in various substrates such as soils and sediments [11–17]. Among the fractionation methods available, Tessier [18] and BCR [19] are some of most cited. Gleyzes [20] reviewed different sequential extraction procedures. However, the oxidisable step of the BCR protocol seemed more effective than that employed in Tessier's method [20]. In order to harmonize sequential extraction procedure, BCR protocol was recommended by Community Bureau of Reference (BCR, now superseded by the Standards, Measurement and Testing Programme) [19]. Recently, because the significant inter-laboratory variability was apparent, in particular in step 2 of the extraction, a thorough re-evaluation of this step of the protocol was conducted [21]. Later, some studies led to the development of a modified BCR sequential extraction procedure [22,23]. The revised protocol involves use of an increased concentration of $\text{NH}_2\text{OH}\cdot\text{HCl}$ and lower pH. It improves reproducibility due, it is thought, to a more efficient dissolution of the reducible fraction of the substrate matrix, most probably the iron oxyhydroxide phase.

MSW is an extremely heterogeneous substrate independent of its geometry, particle size or chemical composition [24]. The high content of organic matter and the heterogeneity of MSW differentiate with soil or sediment significantly. There are no standard fractionation methods for heavy metal and reference samples for MSW till now.

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In some published studies in this field, fractionation of heavy metal was always focused on leachate sediment [5] or municipal solid waste incineration ash [25–27]. Flyhammar [24,28–30] analyzed the fractionation of heavy metals in some specific MSW samples with Tessier sequential extraction procedure, which has been suffering suspicion more and more at oxidisable step. It seems hard to assess the mobility of heavy metal in MSW landfill more effectively.

In the present work, the modified BCR sequential extraction procedure was applied to a variety of MSW samples from landfill site with different degradations for fractionation of copper and zinc, the two elements with the highest level among all heavy metals in MSW. It aims to assess the potential mobility of heavy metal in MSW pertinently.

2. Experimental

2.1. Sampling and pre-treatment

The MSW samples in the experiment were all collected from Tianziling MSW landfill site in Hangzhou, Zhejiang, east China. The Tianziling MSW landfill site, whose design capacity is 6,000,000 m³, was started out business since April 1991. Its bottom liners located at 54 m above sea level and the top of the landfill will reach 165 m above sea level. The matured refuse was located at bottom deposit layers and the fresh one at top deposit layers. Sample A was 0 year aged refuse, which was fresh MSW and sampled from a temporary dumping tank at the top layer of Tianziling landfill site. The other four MSW samples were sampled from four different deposit layers with different depths of the landfill in a sampling well drilled in landfill with the depths of 3 m, 13 m, 25 m, and 40 m below top layer respectively. The four sampling layers were with the deposit ages of 0.5(B), 1(C), 3(D) and 7(E) year respectively.

Five MSW samples were immediately placed in an airtight plastic bag after being sampled. Then, all MSW samples were stored frozen up until analysis. Larger inert objects (including stones, pieces of brick, concrete and cinders) were removed before analysis, and then each sample was quartered to ~500 g sub-samples. A part of them (~200 g) was used to determine the pH, biodegradable matter, moisture and ash content. Samples were dried at 105 °C in a ventilated drying box until a constant weight was achieved. The ash content was determined by burning the dried sample in an oven at 550 °C for 2 h. The pH was measured in 1:5 (w/w) suspensions using a pH-meter (DELTA 320). The biodegradable matter was conducted according to standard methods. The rest of the sample (~300 g) was air dried at <30 °C for 15 days. Subsequently, all air-dried samples were grinded and sieved through a 1 mm nylon mesh. In order to get a representative refuse sample, material under mesh was then homogenized with two steps. First, each of grinded and sieved air-dried sample was filled into a flask, which was manually shaken to rehomogenize them as possible. Sequentially, sample was then moved out from flask and placed on a clean watertight board with a shape of taper. A clean crisscross board was cut from the top of the taper sample, and the sample was separated into four parts. The two diagonal parts were mixed again and quartered as the above process several times till the sample was about 5 g. Finally, the homogenized sub-samples were removed for sequential extraction (1 g) and aqua regia digestion (1 g) respectively.

2.2. Apparatus

Sequential extraction of heavy metals in MSW was performed in 100 ml centrifuge tubes. Quantification was with respect to reagent-matched multi-element standards prepared by serial dilution of 1000 µg ml⁻¹ commercial standard solutions (Guaranteed Reagents). Heavy metals were determined by atomic absorption spectrophotometer (SHIMADZU AA-650).

2.3. Reagents

Extractants were prepared according to the following procedure. All dissolutions and dilutions were performed with distilled water.

Solution A (acetic acid, 0.11 mol l⁻¹): 25±0.2 ml of redistilled glacial acetic acid was added, in a fume cupboard, to about 0.5 l of water in a 1 l polyethylene bottle and made up to exactly 1 l with further water. 250 ml of this solution (0.43 mol l⁻¹ acetic acid) was diluted to 1 l to obtain an acetic acid concentration of 0.11 mol l⁻¹.

Solution B (hydroxylamine hydrochloride, 0.5 mol l⁻¹, pH 1.5): 34.75 g of hydroxylamine hydrochloride was dissolved in 900 ml of water. The solution was acidified with concentrated nitric acid to pH 1.5 and made up to 1 l. This solution was prepared on the same day as the extraction was carried out.

Solution C (hydrogen peroxide, 8.8 mol l⁻¹): hydrogen peroxide was used as supplied by the manufacturer i.e. acid-stabilized to pH 2.0–3.0.

Solution D (ammonium acetate 1.0 mol l⁻¹): 77.08 g of ammonium acetate was dissolved in 900 ml of water. The solution was acidified to pH 2.0 with concentrated nitric acid and made up to 1 l.

2.4. Procedures

2.4.1. Sequential extraction

Sequential extraction was performed using the modified BCR protocol recommended in studies [22,23]. Extractions were performed using the reagents given in Section 2.3 (Table 1). Sequential extraction procedures were described below in detail.

Step 1: A total of 40 ml of solution A (acetic acid) was added to 1 g air-dried MSW sample in a 100 ml centrifuge tube and shaken for 16 h at room temperature (overnight). No delay occurred between the addition of the extractant and the shaking beginning. Finally, the extractant was separated from the solid residue by centrifugation (5000×) and decantation of the supernatant liquid into a high density polyethylene container. The container was stoppered and the extract either analysed immediately or stored at 4 °C. In order to keep the sample similar with the original sample in next extraction procedure as possible, the residue was washed by adding 20 ml of water, shaking for 15 min and finally centrifuging the resulting suspension (5000×). The supernatant was decanted and discarded, taking care not to discard the solid residue. This washing process was conducted three times for each sample. The residue obtained upon centrifugation was broken manually using a vibrating rod prior to the next step.

Step 2: 40 ml of solution B (hydroxylammonium chloride) was added to the residue from Step 1 in the centrifuge tube, and the extraction was performed as described above, especially for residual washing.

Table 1
Schemes of original BCR and modified BCR sequential extraction procedure

Extraction step	Reagents		Fraction
	Original BCR	Modified BCR	
1	0.11 mol l ⁻¹ CH ₃ COOH	0.11 mol l ⁻¹ CH ₃ COOH	Acid soluble
2	0.1 mol l ⁻¹ NH ₂ OH · HCl at pH2.0	0.5 mol l ⁻¹ NH ₂ OH · HCl at pH1.5	Reducible
3	8.8 mol l ⁻¹ H ₂ O ₂ followed by 1.0 mol l ⁻¹ CH ₃ COONH ₄ at pH 2	8.8 mol l ⁻¹ H ₂ O ₂ followed by 1.0 mol l ⁻¹ CH ₃ COONH ₄ at pH 2	Oxidable
Residual	Aqua regia	Aqua regia	Residual

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