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# Evaluation of Sediments Decontamination by Chelating Agents using Thermogravimetric Analysis

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**ABSTRACT:** This study investigated single-step extraction of heavy metals from contaminated sediments collected in the port of Cádiz. Experimental tests of washing with an aqueous solution of a chelating agent were performed at selected solid/liquid (S/L) ratios. Two chelating agents were chosen: 0.3 M EDTA (pH 3.8 and 8) and sodium citrate (1 M). The objective of the tests was to investigate extractive decontamination by the washing treatment. The study was focused on the extraction of Pb, Cu, Zn, and Cd. The experiments showed that the decontamination efficiencies of the extractants were of the order: EDTA (pH 3.8) > EDTA (pH 8) ~ sodium citrate. EDTA solution with slightly acid pH was optimal for the extraction of Pb, Cu, Zn and Cd. The data obtained from the thermal analysis of sediments after the decontamination process indicated the success of the extraction procedure which is proposed as a technique for controlling the behaviour of the decontamination process.

**Key words:** Cádiz port, Chelating agent, Heavy metals, Decontamination, Thermal analysis

## INTRODUCTION

Marine sediments comprise a complex, heterogeneous mixture of geochemical phases able to bind anthropogenic heavy metals by precipitation, ion exchange and adsorption (Allen, 1993). These phases include readily exchangeable forms such as carbonate, hydrous iron, manganese oxide, as well as organic matter and sulphides, and the strength with which heavy metals bind to these phases determines their relative bioavailability (Tessier *et al*., 1979). Heavy metal contamination primarily originates from several industrial and urban sources along the coastal regions. The predominant mode of heavy metal retention in sediments can be significantly influenced by the level of sediment contamination by the metal, in addition to geochemical factors and behaviours of the metal itself. In Cádiz Port the main industries located in this zone are associated with ship construction and boat maintenance activities, aerospace component manufacturing, power generation, and marine transport. All these activities have a negative environmental impact and have led to an increase in pollution of marine sediments in this zone. Many studies of contaminants in the sediments in this zone (Ligero *et al*., 2002; Carrasco *et al*., 2003 and Rodríguez-Barroso *et al*., 2006, 2008) have shown contamination levels between low and moderate, and so there is a need to perform experimental tests to evaluate the possibility of decontaminating these sediments. The technologies used for sediment decontamination are similar to those generally adopted for soil (Peters and Shem, 1995), including a washing process that separates the finer soil particles which frequently contain a greater percentage by weight of the metal contaminant. This can be followed by extractions using acidic solutions, chelating agents, oxidising/reducing agents, or other chemical treatments to render the metal more extractable (Ahmad *et al*., 2005).

In the *ex situ* washing technology (Freeman *et al*., 1995; Moutsatsou *et al*., 2006) undesirable contaminants in soils or sediments are removed by dissolving or suspending them in a washing solution. In the case of soils or sediments contaminated with heavy metals, the washing solution is generally an aqueous solution of a chelating agent such as ethylenediamine tetraacetic acid (EDTA), nitriloacetic acid (NTA), diethylenetriamine pentaacetic acid (DTPA), S,S-ethylene-diamine-disuccinic acid (EDDS) and sodium citrate.

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EDTA is one of the most widely used complexing agents because of its high extraction capacity (Sahuquillo *et al.*, 2003); it has been widely used in agronomy for estimating total extractable metal in soils (Alvarez *et al.*, 2006; Manouchehri *et al.*, 2006). EDTA is assumed to extract metals on exchange sites of both inorganic and organic complexes. Additionally, it can dissolve calcareous materials through complexation of calcium and magnesium (Chao, 1984; Gismera *et al.*, 2004; Sahuquillo *et al.*, 2003). Another study has reported the use of EDTA to remove metals which are organically bound, occluded in oxides, or associated with secondary clay minerals (Payà-Pérez *et al.*, 1993). In our laboratory studies have shown that EDTA is effective in removing Pb, Zn, Cu and Cd from contaminated sediments, although the extraction efficiency depends on many factors such as the strength of the EDTA and the pH (Oudghiri *et al.*, 2011a).

In this paper experimental tests of metal extraction from contaminated sediments are presented and discussed. Sediments were collected from Cádiz port, where significant heavy metal content has been observed. In particular, Pb, Cu, Zn and Cd were chosen as target pollutants in this study. For this purpose the aims of this study were to compare the extractability of trace metals in Cádiz harbour sediments using $Na_2$-EDTA (pH 3.8 and 8) and sodium citrate. Additional objectives were to determine whether extractability using EDTA correlated with the results of thermogravimetric analysis (TGA).

## MATERIALS & METHODS

The sediments used in this study were collected in an industrial harbour in Cádiz, Spain (S1 was collected at 8.6 meters depth and S2 at 12.2 meters). Sediments were characterised by several standard methods: grain size distribution was carried out by wet sieving of the 63 m particle size fraction (Rodier, 1997); organic matter was evaluated by calcination for 2 h at 550ºC according to DIN 38 414-S3 (1985); carbonate content ($CaCO_3$) was determined using a Bernard calcimeter by the acid leaching method (Wiesmann and Nehring, 1951); the concentrations of the elements Mn, Cr, Cu, Ni, Zn, Pb, Cd and Hg were measured with ICP-MS (inductively coupled plasma mass spectrometry) and those of Fe were determined with ICP-AES (inductively coupled plasma atomic emission spectroscopy) after digestion of the sediment according to Förstner and Wittmann (1983). The analytical procedure was previously validated by chemical analyses of standard sediments (MESS-3 and PACS-2).

The extractive decontamination tests were performed at room temperature for 24 h. For this, two sediment samples (S1 and S2) were treated with two aqueous solutions of chelating agent at a selected 1:10 solid/liquid ratio: (1) $Na_2$EDTA (0.3 M at pH 3.8 and 8); (2) sodium citrate (1 M). The pH was adjusted to pH 3.8 and 8 using small volumes of HCl or NaOH (both 1 M). The suspensions were centrifuged at 4000 rpm for 15 min and the resulting mixture was filtered at 0.45µm. The concentrations of metals were determined by ICP-AES (Thermo Elemental IRIS Intrepid) or by ICP-MS (Thermo Elemental Series X7). The sediment residues were rinsed with distilled water to remove the residual extraction, dried for 1 h at 105ºC, and ground before TGA analysis. Thermal behaviour was analysed through thermogravimetric analysis (TGA) using a simultaneous thermal analyser (Perkin Elmer STA 6000). For these experiments, approximately 10 mg of sediment was placed in a ceramic crucible; the TGA operating conditions were temperature ramps at 10ºC/min from 100 to 900ºC. The analyses were conducted under a nitrogen (pyrolysis) atmosphere with a 25 mL/min gas flow, and the percentage mass loss was calculated according to the equation below:

$$\text{Weight loss (\%)} = \left(\frac{m_i - m_a}{m_i}\right) \times 100$$

where $m_i$ is the initial mass (milligram) and $m_a$ is the actual mass (milligram).

## RESULTS & DISCUSSION

The chemical and physical properties of the sediments are set out in Table 1. According to the textural analysis, sediment S1 was more fine-grained than sediment S2, which contained similar mud and sand fractions. Organic matter and $CaCO_3$ content were higher in sediment S1 than in S2. Total concentrations of Pb, Zn, Cu and Cd in these two areas exceeded the maximum level of reference background (Turekian and Wedepohl, 1961): Pb d” 20 mg/kg, Zn d” 95 mg/kg, Cu d” 45 mg/kg, Cd d” 0.3 mg/kg, Fe d” 47000 mg/kg, Mn d” 850 mg/kg, and according to previous work (Oudghiri *et al.*, 2014) showed a higher pollution load index (PLI) for samples studied, indicating that metal pollution in these two study areas is very serious.

Fig. 1 illustrates the trend of Pb, Cu, Zn and Cd extraction yield, calculated with respect to the total initial amount for tests of sediment S1 and S2, using EDTA 0.3 M (pH 3.8), EDTA 0.3 M (pH 8) and sodium citrate. As seen in Fig. 1, the extraction efficiencies for Pb, Cu, Zn and Cd with EDTA 0.3 M pH (3.8) were always higher than those for EDTA 0.3 M (pH 8) and sodium citrate in the both samples. These results indicate that EDTA is an effective extractant because of its strong ability to chelate various heavy metals. The variation of extraction between EDTA pH 3.8 and

**Table 1. Physicochemical characteristics of the sediments**

| Sediments | Fraction < 63 µm (Wt %) | Fraction > 63 µm (Wt %) | Organic matter (%) | $CaCO_3$ (%) | Total metals (mg/kg) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Pb | Cu | Zn | Cd | Fe | Mn |
| S1 | 87.98 | 12.02 | 12.37 | 30.89 | 75.08 | 186.11 | 261.55 | 0.51 | 34374 | 439 |
| S2 | 56.91 | 43.09 | 9.45 | 24.77 | 93.65 | 124.54 | 281.47 | 0.44 | 33608 | 488 |

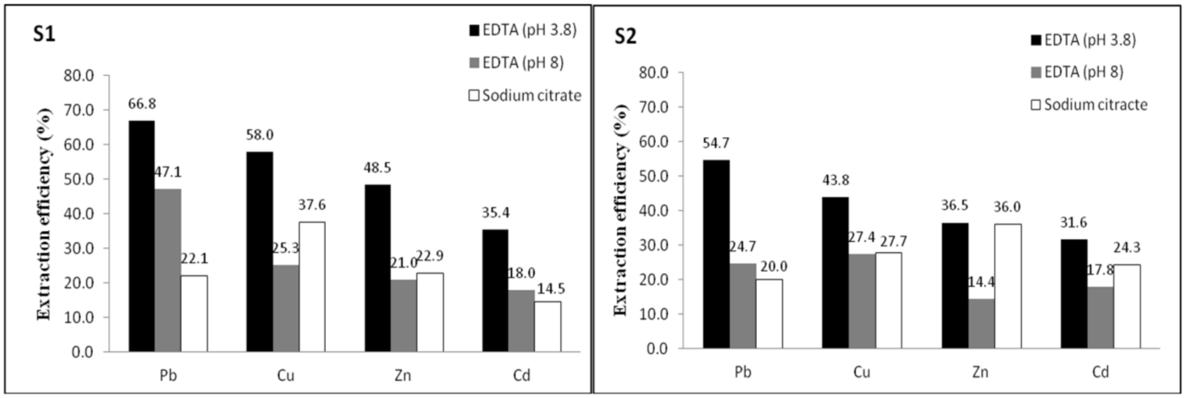

**Fig. 1. Result of sediments extraction tests**

EDTA pH 8 can be explained by the influence of pH on the extraction of heavy metals. Previous studies showed that pH influenced the extraction of heavy metals by EDTA only in the acidic range (pH < 5) (Elliott and Brown, 1989; Yu el al., 1994; Oudghiri *et al.*, 2011b). The removal of greater amounts of toxic metals has most often been observed at lower pH levels (van Benschoten *et al.*, 1997). However, other authors have reported that a slightly alkaline pH was optimal for the removal of Pb, Zn and Cd with [S,S]-EDDS (Vandevivere *et al.*, 2001). The average extraction efficiencies for Pb, Cu, Zn, and Cd in sediment S1 by EDTA 0.3 M pH (3.8) were 66.8%, 58%, 48.5% and 35.4%, respectively, while in sediment S2 they were 54.7%, 43.8%, 36.5% and 31.6% respectively. However, a general difficulty in cadmium extraction was observed in both cases due to the high stability of cadmium humate. In addition, the percentage extractabilities of metal followed the sequence of Pb > Cu > Zn > Cd; this is likely to be related to the strong complexing capacity of EDTA with respect to heavy metals and the formation of the stable 1:1 metal–EDTA complexes, the formation constants of which could reach 19.0, 19.7, 17.5 and 17.4 for Pb-EDTA, Cu-EDTA, Zn-EDTA and Cd-EDTA, respectively (Sun *et al.*, 2001). The formation of complexes in sediments is controlled by the kinetics of all complexation reactions, adsorption in sediment solid phases, mineral dissolution, and the possible degradation of the chelating agent or its metal complexes (Nowack, 2002). These interactions are difficult to predict and depend on the contaminants and sediment conditions. The amounts of Zn, Cd, Cu and Pb extracted with 0.3 M EDTA (pH 3.8) in a ratio of 1:10 (S/L) varied widely between sediments S1 and S2; as seen in Fig. 1, the extraction efficiency of Pb, Zn, Cu and Cd in sediment S1 was higher than that in sediment S2. This variation may have two possible reasons. On one hand, the sediment S2 is highly contaminated and contained far more heavy metals than the amount of EDTA added (Table 1), resulting in almost complete complexation of EDTA by heavy metals; therefore, it is easier for metals in sediment S1 to be extracted by EDTA than those in sediment S2. On the other hand, the higher the proportion of coarse particles in sediment, the lower the efficiency of EDTA extraction, because extracted heavy metals could be easily adsorbed by iron–manganese oxides located on the surface of those sediment particles. The amounts of heavy metals adsorbed on sediment particles could be influenced by many other factors, such as the amounts of sediment, pH, reagent concentration, water losses and the heavy metal content in the sediments, and were closely related to the content of sediment particles 63 µm in size (Szava-Kovats, 2008). The TGA (%) and the differential thermogravimetric curve (DTG, % $min^{-1}$) of sediment untreated and treated with extractant at the heating rate of 10ºC/min are presented in Fig. 2. The weight losses, shown in Table 2, correspond to the main peaks shown in the thermograms and their corresponding temperature ranges, in sediments S1 and S2 before (untreated) and after extraction. Three stages of thermal decomposition were found in the interval from 100 to 900ºC as shown. The first stage (100 to 200ºC) was attributed to the chemically bound

**Table 2. Relative weight losses in selected temperature intervals during thermogravimetric analysis (TGA) analyses**

| | **$\Delta W_1$ (%)** | **$\Delta W_2$ (%)** | **$\Delta W_4$ (%)** | **$\Delta W_{Total}$ (%)** |
|---|---|---|---|---|
| | **(100–200°C)** | **( 200–600°C)** | **(600–900°C)** | **(100–900°C)** |
| **S1 untreated** | 0.48 | 6.86 | 13.27 | 20.60 |
| **EDTA (pH 3.8)** | 0.44 | 6.59 | 1.97 | 9.00 |
| **EDTA (pH 8)** | 0.43 | 6.23 | 8.70 | 15.37 |
| **Sodium citrate** | 0.39 | 6.52 | 10.09 | 16.91 |
| **S2 untreated** | 0.41 | 6.13 | 11.11 | 17.65 |
| **EDTA (pH 3.8)** | 0.40 | 6.02 | 3.37 | 9.79 |
| **EDTA (pH 8)** | 0.40 | 6.13 | 6.76 | 13.29 |
| **Sodium citrate** | 0.41 | 5.79 | 7.87 | 14.07 |

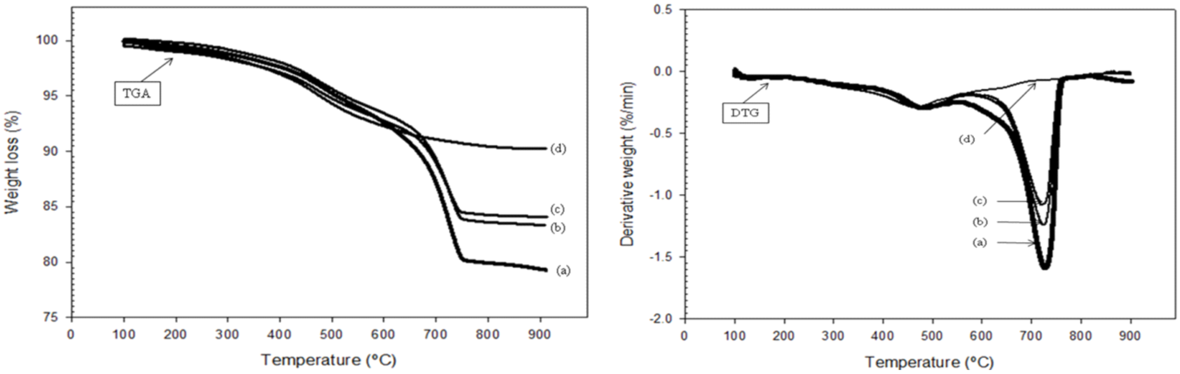

**Fig. 2. Thermogravimetric analysis (TGA) and differential thermogravimetric curve (DTG) of sediment S1 (a) untreated, and treated with (b) sodium citrate, (c) EDTA (pH 8) and (d) EDTA (pH 3.8)**

water and hygroscopic water of salts and organic matter (Schnitzer *et al*., 1964; Liptay, 1974; Gaál *et al*., 1994). The second stage (200 to 600°C) was due mainly to the decarboxylation of organic matter and dehydroxylation of clay minerals (Lopez-Capel *et al*., 2006a; Plante *et al*., 2005). The last stage (600 to 900°C) corresponds to carbonate-associated inorganic compounds (Oudghiri *et al*., 2014; Lopez-Capel *et al*., 2006b).

The results showed that the total weight loss ($DW_{total}$, %) of treated sediments were lower than that in untreated sediments (Table 2). Therefore, the weight losses of sediment S1 untreated (20.60%) were higher than the same treated with sodium citrate (16.91%), followed by EDTA at pH 8 (15.35%), and finally EDTA pH at 3.8 (9.00%); the behaviour was the same for S2. These differences may be explained by the extraction of metal during the decontamination process, resulting in a lower metal content compared with its initial composition. These results are in good agreement with the decontamination efficiencies of the extractants being in the order: EDTA (pH 3.8) > EDTA (pH 8), with sodium citrate being very similar to the latter.

In addition, with respect to the different temperature ranges (shown in Fig. 2 for sediment S1), the treatment significantly reduced the weight losses observed in the range 600–900ºC and had little influence on the weight loss in the temperature range 200–600ºC; this indicates the presence of organic matter which is more thermally stable during thermal decomposition, indicating that this content was not really affected by our extraction process.

EDTA extraction under acidic conditions (pH 3.8) destroyed the carbonate, as confirmed by a decrease in the weight loss in the range 600–900ºC: 1.97% versus 13.27% in the untreated sample S1 and 3.37% versus 11.11% in the untreated sample S2. Furthermore, these results have demonstrated that the extraction of Pb, Cu, Zn and Cd from the carbonate fraction of the sediment was almost total, a result in accordance with the important increases in extraction efficiencies for sediment due to EDTA at pH 3.8. Overall, the results from the thermogravimetric techniques indicated the success of the extraction procedure.

## CONCLUSIONS

This study investigated the extractive decontamination of Pb, Cu, Zn and Cd from contaminated sediment using EDTA (pH 3.8 and 8) or sodium citrate. The results of the experimental tests performed at different extraction conditions and 1:10 solid/liquid ratio showed clearly that EDTA at pH 3.8 was an effective extracting agent because of its strong chelating affinity for different heavy metals; the percentage extractabilities of metal followed the sequence Pb > Cu > Zn > Cd. In view of the field application of the washing technology for sediment decontamination, the single-step extraction using EDTA under acidic conditions was found to be a useful method to evaluate extraction of heavy metals. The data obtained from the thermal analysis of sediments after the decontamination process indicated the success of the extraction procedure which is proposed as a technique for controlling the behaviour of the decontamination process

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