

Adsorption of Pb(II) on Spent Leaves of Green and Black Tea

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Abstract: Problem statement: In recent years much attention has been focused on the use of biomass residues as low-cost adsorbents for the removal of heavy metal ions from contaminated waters. Spent tea leaves, an abundantly available material that is currently disposed of as a solid waste, are potentially suitable for such applications. **Approach:** To provide some information on the adsorption properties of tea waste, we evaluated the removal efficiency of lead ions by spent leaves of green and black tea. Batch adsorption experiments were made at 25 and 40°C at initial lead-ion concentrations between 0.01 and 2 g L⁻¹. Equilibrium data were analyzed by the Langmuir equation to evaluate the maximum adsorption capacity and the equilibrium constant. The adsorption characteristics of the two materials were also compared with those of coffee grounds, activated carbon and Fuller's earth. **Results:** Experimental data showed that removal efficiencies up to 98-99% can be achieved when using spent tea leaves as lead adsorbent. The results were only marginally affected by the type of tea waste. At low lead loading, the adsorption equilibrium was well described by the Langmuir equation, with a maximum adsorption capacity of 83-130 mg g⁻¹ and an equilibrium constant ranging from 0.112-0.245 L mg⁻¹. A comparison with other adsorbents provided the following order for lead removal efficiency: Black tea, coffee grounds > green tea > Fuller's earth > activated carbon. **Conclusion:** The results from this study indicate that using spent tea leaves as an adsorbent may be an efficient and economical means for removing lead and, presumably, other heavy metal ions from aqueous solutions.

Key words: Lead, adsorption, spent tea leaves, black tea, green tea

INTRODUCTION

Heavy metals, such as cadmium, chromium, lead and mercury, are highly toxic pollutants of increasing concern in developed countries for their significant impact on the environment and human health (Coen *et al.*, 2001; Arowolo, 2004; Giller *et al.*, 2009). Lead (Pb), in particular, is one of the most common environmental contaminants due to its wide use in petroleum, mining, paint and pigments, ceramics and weapons industries. Its accumulation in the body may cause several pathological states, including brain damage, kidney failure and serious developmental, learning and behavioral problems in children (Needleman, 2004; Bellinger, 2008; Rowland and McKinstry, 2006). In addition, toxicity has been shown to occur at concentrations as low as 1 pM (Markovac and Goldstein, 1988; Johnston and Goldstein, 1998). For these reasons, over the past few years the allowable levels of lead in drinking and

irrigation waters have undergone major reductions in most countries.

Adsorption of heavy metal ions onto activated carbon is an efficient and well established method for their removal from contaminated waters, but high costs limit its widespread use. Thus, in recent years much research has been undertaken to develop comparably effective but less expensive adsorbents (Bailey *et al.*, 1999; Babel and Kurniawan, 2003; Demirbas, 2008). An economical adsorbent is defined as one which is abundant in nature, or is a by-product or a waste from industry and requires little processing (Aksu and Isoglu, 2005).

Given the above considerations and requirements, we choose to explore the suitability of spent tea leaves as an adsorbent for the removal of lead ions from aqueous solutions. After water, tea is the most widely consumed beverage in the world, as attested by the over 3,000,000 tons of tea leaves produced annually (Wan *et al.*, 2008). Although available in different varieties, such as green,

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black or Oolong tea, all tea beverages are obtained from the same basic tea (*Camellia sinensis* L.) leaves (Wan *et al.*, 2008). Once the beverage has been brewed, spent leaves become a waste that must be disposed of. Like other biomass residues, tea wastes represent an unused resource and pose increasing disposal problems (Arvanitoyannis and Varzakas, 2008). Some studies have demonstrated the ability of tea wastes to remove synthetic dyes (Hameed, 2009), turbidity (Sabrina and Hasmah, 2008) and even some types of heavy metal ions (Mahvi *et al.*, 2005; Amarasinghe and Williams, 2007; Sabrina and Hasmah, 2008) from water. However, there appears to be a large variability in the rate and degree of adsorption, depending on the nature of the contaminant, the presence of other competitive species and the process conditions. Such variations require careful analysis of the properties of the adsorbent at the specific conditions under which it will be used.

In this study we present the results of an experimental study aimed at a preliminary assessment of the adsorption characteristics of lead ions on spent leaves of black and green tea. Lead adsorption was investigated in a batch system over a wide range of initial lead-ion concentrations. The performance of tea waste was compared to that attained with a similar biomass residue, coffee grounds and two commonly used adsorbents, activated carbon and Fuller's earth.

MATERIALS AND METHODS

Chemicals: Pb(II) chloride with a purity >98% was obtained from Sigma-Aldrich (Milano, Italy). Granular activated carbon (8-mm pellets) and powdered Fuller's earth were from Sigma-Aldrich (Milano, Italy). All other chemicals were of analytical grade and used without further purification.

Analytical methods: Lead-ion concentration in the aqueous solutions was determined colorimetrically by a standard test (Nanocolor[®] Lead-5, Macherey-Nagel GmbH, Germany) based on the reaction of Pb(II) with 4-(pyridyl-2-azo)-resorcinol.

Tea waste preparation and characterization:

Samples of tea waste were obtained from commercial green and black tea leaves. Beverages were first prepared according to package instructions suggested by the manufacturers. Typically, one tea bag was steeped, under gentle stirring, in 125 mL of distilled water at 90°C for 3 min. After this time, spent tea leaves were recovered and repeatedly washed with distilled water in order to remove soluble and colored compounds. Then the solid was rinsed with distilled

water and oven dried at 60°C for 24 h. Finally, the dried leaves were ground and sieved to particles <500 µm which were stored in polyethylene bags at room temperature until use.

The tea waste was analyzed for moisture, fiber, protein, fat, wax, ash and ash alkalinity, according to official analytical methods.

Equilibrium studies: Batch adsorption experiments were carried out in 50 mL screw-top pyrex flasks. The flasks were placed in a water bath thermostated at 25 or 40 (±0.05)°C and were magnetically stirred. In a typical experiment, about 0.2 g of dried leaves were contacted with 40 mL of an aqueous solution of lead chloride at pH 5.5. Preliminary runs showed that equilibrium was reached in 18-20 h. Accordingly, the duration of these runs was set at 24 h. After this time, the stirring was stopped and a sample of the solid-liquid suspension was taken. The suspension was filtered two times on filter paper and the liquid was analyzed for lead content. The initial lead concentration was varied between 0.01 and 2 g L⁻¹ and the solid to liquid ratio between 1 and 10 g L⁻¹.

Comparison of adsorbents: In these experiments about 0.5 g of the adsorbent were contacted with 50 mL of an aqueous solution at 0.1 g L⁻¹ lead concentration. The contact time was set at 2 h and the temperature at 25 or 40°C. In addition to green and black tea leaves, the following adsorbents were used: Coffee grounds, activated carbon and Fuller's earth.

RESULTS

The physicochemical characterization of tea waste samples gave the results presented in Table 1. As can be seen, the waste was rich in crude fiber (36.2% w/w) and proteins (22.6%). In contrast, fats amounted to slightly more than 1% by weight. Waxes were made up of esters with 34-46 carbon atoms and the compound with 34 carbon atoms was the most abundant.

The percent lead removal efficiency was expressed as:

$$R\% = \frac{c_0 - c_f}{c_0} \times 100 \quad (1)$$

where, c_0 and c_f are the initial and final concentrations of lead (mg L⁻¹) in the aqueous solution.

A preliminary evaluation of the effect of Solid-to-Liquid ratio (S/L) on R% provided the trend shown in Fig. 1, indicating that for S/L >5 g L⁻¹ the lead removal efficiency was essentially independent of this ratio.

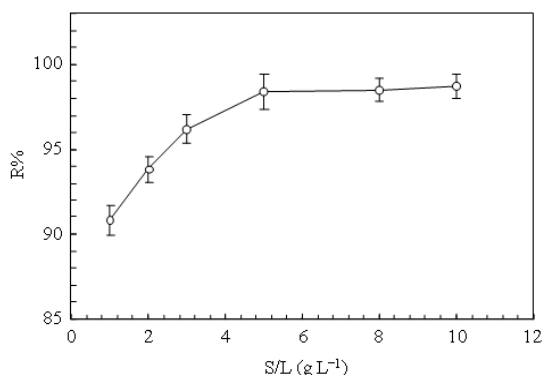


Fig. 1: Effect of Solid-to-Liquid ratio (S/L) on the percent lead removal efficiency (R%) at 25°C using spent leaves of green tea as adsorbent

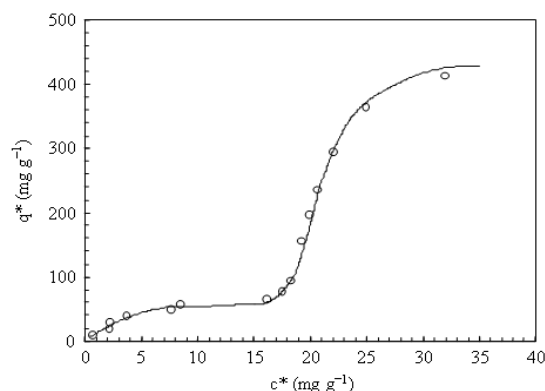


Fig. 2: Equilibrium data for lead adsorption on spent leaves of green tea at 25°C.

Table 1: Physicochemical properties of tea waste

Parameter	Method	Value	Unit
Moisture	ISTISAN 96/34 p. 7	6.21±0.23	w/w %
Crude fiber	ISTISAN 96/34 p. 68	36.20±0.8	w/w %
Proteins	ISTISAN 96/34 p. 13	22.60±0.3	w/w %
Fats	ISTISAN 96/34 p. 39	1.20±0.2	w/w %
Total waxes	GU CE L161 22/06/2007	73.00±16	mg kg ⁻¹
C-34 ester	GU CE L161 22/06/2007	44.00±9.6	mg kg ⁻¹
C-36 ester	GU CE L161 22/06/2007	17.00±3.8	mg kg ⁻¹
C-38 ester	GU CE L161 22/06/2007	2.00±0.4	mg kg ⁻¹
C-40 ester	GU CE L161 22/06/2007	2.00±0.4	mg kg ⁻¹
C-42 ester	GU CE L161 22/06/2007	2.00±0.4	mg kg ⁻¹
C-44 ester	GU CE L161 22/06/2007	1.00±0.2	mg kg ⁻¹
C-46 ester	GU CE L161 22/06/2007	5.00±1.1	mg kg ⁻¹
Ash	ISTISAN 96/34 p. 77	6.22±0.05	% w/w
Ash alkalinity	DM 03/02/1989 II.14	888.00±30	mL HCl / 1 M/100 g

Accordingly, batch adsorption experiments were carried out at S/L = 5 g L⁻¹ or higher. In these experiments, the specific amount of lead adsorbed at equilibrium, q* (mg g⁻¹), was calculated by:

$$q^* = \frac{V_L}{w} (c_0 - c^*) \quad (2)$$

Where:

c₀, c* (mg L⁻¹) = The liquid-phase concentrations of lead at initial and equilibrium, respectively

V_L(L) = The volume of the solution
w(g) = The dry mass of adsorbent

Figure 2 displays the results obtained at 25°C for lead adsorption on spent leaves of green tea. A similar trend was observed when using black tea leaves. Data points in the Fig. 2 indicate the occurrence of multilayer adsorption, with a first-layer adsorption capacity below 100 mg g⁻¹ and the second of the order of 450-500 mg g⁻¹.

Since in practical applications the solutions to be treated are generally highly diluted in lead, we examined more closely and quantitatively the adsorption properties of tea wastes at low initial lead concentrations (c₀ < 500 ppm, which roughly corresponds to c* < 20 mg L⁻¹). For both materials, the adsorption behavior was found to be well described by the Langmuir equation:

$$\frac{q^*}{q_m} = \frac{K_L c^*}{1 + K_L c^*} \quad (3)$$

Where:

q_m = The maximum adsorption capacity
K_L = The adsorption equilibrium constant

These parameters were evaluated by minimizing the following objective function:

$$\Phi(K_L, q_m) = \sum_{i=1}^n (q_{exp,i} - q_{calc,i})^2 \quad (4)$$

representing the sum of squared errors between experimental and calculated lead concentrations in the solid. The estimates of K_L and of q_m are listed in Table 2, while a comparison between experimental data and model results is shown in Fig. 3 and 4.

The percent lead removal efficiencies at high lead loading are reported in Fig. 5. The values of R% were very high (between 94.4 and 98.6%) and increased with the concentration of lead ions in the liquid. Furthermore, there appeared to be only small differences (<1%) between green and black tea wastes, the former being slightly more effective.

Table 2: Langmuir adsorption parameters (q_m , K_L) for lead adsorption on green and black tea wastes. c_0 is the initial concentration of lead ions in the aqueous solutions

Waste material	T (°C)	Δc_0 (mg L ⁻¹)	q_m (mg g ⁻¹)	K_L (L mg ⁻¹)
Green tea	25	149-400	90.1	0.173
	40	141-358	83.3	0.245
Black tea	25	70-351	129.9	0.112
	40	137-342	101.0	0.223

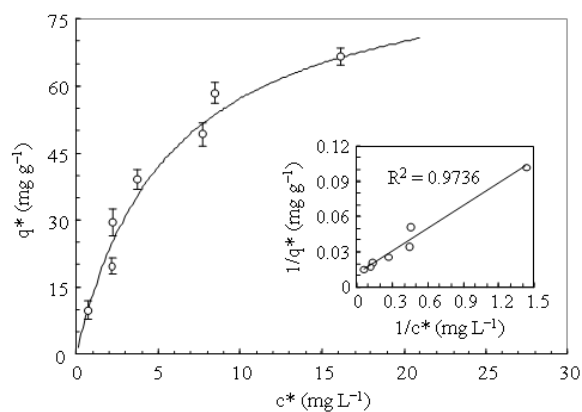


Fig. 3: Experimental data and Langmuir isotherm for lead adsorption on spent leaves of green tea at 25°C

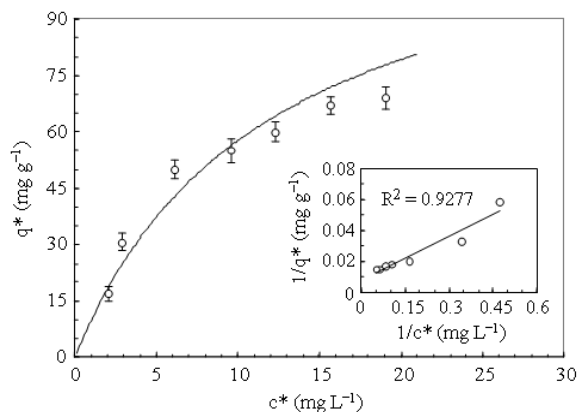


Fig. 4: Experimental data and Langmuir isotherm for lead adsorption on spent leaves of black tea at 25°C

Finally, the comparison of adsorbents gave the results in Table 3. Since the materials examined (spent tea leaves, coffee grounds, activated carbon and Fuller's earth) had different moisture contents, we also calculated the amount of lead adsorbed per unit weight of dry solid (q_s), obtaining the results reported in Fig. 6. Examination of these results reveals two main points: (a) temperature has a limited influence on adsorption,

Table 3: Lead removal efficiency (R%) and moisture content (U%) of tea wastes and other adsorbents

Adsorbent	U%	R%	
		T = 25°C	T = 40°C
Black tea waste	11.6	98.4	99.3
Green tea waste	7.68	98.0	98.0
Coffee grounds	10.95	99.0	99.0
Activated carbon	2.18	99.3	97.9
Fuller's earth	2.94	99.4	99.4

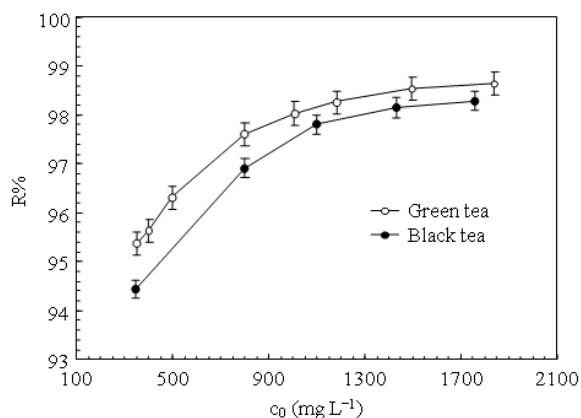


Fig. 5: Percent lead removal efficiencies (R%) at high lead loading on spent leaves of green and black tea at 25°C. c_0 is the initial concentration of lead ions in the aqueous solutions.

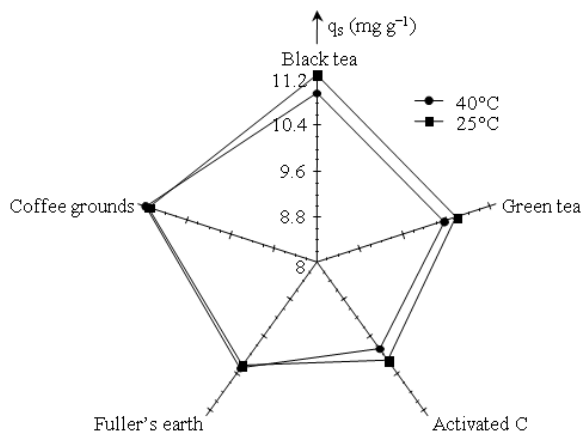


Fig. 6: Comparison of adsorbents for lead removal efficiency at 25 and 40°C. q_s is the amount of lead adsorbed per unit weight of dry solid

at least between 25 and 40°C; (b) green and black tea wastes are, together with coffee grounds, the most effective adsorbents. Overall, the order of lead removal efficiency for the various materials was as follows: Black tea waste, coffee grounds > green tea waste > Fuller's earth > activated carbon.

DISCUSSION

The results from the experiments described in this study show that lead ions adsorb efficiently on spent tea leaves and that limited differences exist between green and black tea. These findings are in agreement with the few reports in the literature that have addressed the possibility of using tea wastes for such purposes (Tee and Khan, 1988; Mahvi *et al.*, 2005; Amarasinghe and Williams, 2007; Sabrina and Hasmah, 2008). In particular, Amarasinghe and Williams (2007) reported percentages of lead-ion removal by spent tea leaves between 37-94%, for a solid-to-liquid ratio ranging from 1.25-7.5 g L⁻¹. A similar behavior was observed for copper ions, even though the affinity for this metal species was lower, with percent removals between 20 and 70%. Fairly high removal efficiencies were also determined for nickel and cadmium, with maximum values of about 86 and 77%, respectively (Mahvi *et al.*, 2005). Experiments on the simultaneous removal of metal ions from aqueous solutions by spent tea leaves provided an evidence for their competitive adsorption (Mahvi *et al.*, 2005; Amarasinghe and Williams, 2007), suggesting that these species are bound by the same surface sites.

It is now well established that the ability of a waste material to bind lead or, more generally, heavy metals is the result of a number of mechanisms, including surface adsorption, chemisorption, complexation, ion exchange, microprecipitation and metal hydroxide condensation (Demirbas, 2008). In order to understand the detailed mechanism by which metals are removed by adsorption, it is essential to identify the functional groups involved in binding. Although specific studies on tea waste are lacking, charged and polar functional groups on the surface of proteins or of other cell-wall components (cellulose, hemicellulose, pectin, lignin) are considered to be the main responsible for metal removal (Basso *et al.*, 2002; Pagnanelli *et al.*, 2003). Metal-binding sites can replace hydrogen ions with metal ions or donate an electron pair to form metal complexes (Ofomaja and Ho, 2007; Demirbas, 2008).

Among metals, lead is known to interact strongly with proteins, this interaction being the basis of the lead-induced cytotoxicity (Goering, 1993). The lead-protein association is mediated by both electrostatic interactions and covalent bonds. The former occur between the positively charged lead ions and some negatively charged groups on the protein surface, while covalent bonds are formed between lead ions and N-, O- or C-protein terminals (Dutta *et al.*, 2005; Ghering *et al.*, 2005; Srinivas *et al.*, 2007). Other potential lead-binding sites are the hydroxyl and

carboxyl groups on the cellulose-based plant fibers (Zhou *et al.*, 2005; Demirbas, 2008). Also the phenolic compounds contained in the tea leaf have a significant affinity for lead ions, which manifests itself in their capability of capturing these metal species from aqueous solutions (Yu *et al.*, 2001). Nevertheless, phenolics are for a large part extracted during brewing and their residual content in the waste presumably provides a minor contribution to the overall binding capacity of spent tea. This holds, in particular, for tannins and tannin derivatives. These compounds are present at high levels in black and green tea but are rapidly released during infusion, being mainly localized on the outside of the leaf. Thus, the high lead removal efficiency determined in our experiments is probably to be ascribed to the large amounts of proteins and fiber contained in the tea waste (Table 1).

The observed multilayer adsorption isotherms, an example of which is shown in Fig. 2, are very similar to those reported by Andini *et al.* (2006), who investigated the adsorption of Pb(II) and Cd(II) onto an organophilic bentonite. This behavior suggests that at low lead concentrations moderate interactions occur between metal ions and tea waste. As adsorption proceeds, the interactions between the adsorbate and the adsorbent increase and cooperatively promote lead loading on the solid. The second-layer adsorption capacity was about 5-to 6-fold higher than that of the first layer, indicating that clusters of lead ions are bound to sites on the surface of tea leaves. Interestingly, the ratio between the two adsorption capacities was very close to those found by Andini *et al.* (2006) for Pb and Cd adsorption on bentonite.

The substantial similarity of green and black tea, both in terms of percent lead removal efficiency (Fig. 5) and of Langmuir parameters (Table 2), can be ascribed to the fact that these materials consist of the same basic tea leaves (*Camellia sinensis* L.), being black tea produced from mildly fermented green tea (Wan *et al.*, 2008). An important consequence is that a mixed waste made up of varying amounts of green and black tea leaves can be expected to be fairly homogeneous in its lead-adsorption properties.

Comparison of adsorbents for lead removal efficiency reveals that spent tea leaves and coffee grounds behave in a quite similar way, with average adsorption capacities between 10.81±0.42 mg g⁻¹ (at 40°C) and 10.99±0.11 mg g⁻¹ (at 25°C). Although these materials differ in their specific chemical composition, the main classes of components are essentially the same, due to their common plant derivation. A study on the removal of lead ions from drinking water performed by coffee grounds showed a strong correlation between

the protein content and the amount of lead adsorbed (Tokimoto *et al.*, 2005). In particular, the removal of lead ions was considerably reduced when the proteins contained in the coffee grounds were subjected to a denaturing treatment with perchloric acid, dropping to zero when the material was boiled in 10% perchloric acid for 10 min. These observations add further support to the hypothesis that, similarly to coffee grounds, the protein fraction of tea waste provides one or perhaps the major contribution to its lead removal capability.

CONCLUSION

The results from this study clearly support the possibility of using spent leaves of green and black tea to remove lead ions from aqueous solutions. Removal efficiencies were found to fall within the range of 94-99%, depending on the solution composition and the adsorption conditions. This material was also more effective than conventional adsorbents such as activated carbon and Fuller's earth.

Tea wastes are produced in nearly all parts of the world and, although of no commercial value, their disposal is costly and not devoid of environmental impact. The utilization of spent tea leaves as an adsorbent of lead or of other metal ions may therefore represent an attractive strategy for the effective reduction and reuse of this type of waste.

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