



Article Removal of Lead by Oxidized Graphite

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Abstract: Lead ion adsorption on the surfaces of pristine and oxidized graphite is studied quantitatively using X-ray photoelectron spectroscopy (XPS) and standard electrochemical measurements. The XPS analysis confirmed the oxidation of graphite, yielding a final composite consisting of 15.97% of oxygen and 84.03% of carbon in comparison with the pristine graphite powder consisting of 6.13% oxygen and 93.87% carbon. The adsorption of lead (II) ion was confirmed by the peaks observed at 138 eV and 143.8 eV, associated with the emissions from Pb4f 7/2 and Pb4f 5/2, respectively. The effective concentration of Pb²⁺ ion and the optimum dosage of oxidized graphite were calculated to be 400 μ M and 200 mg, respectively. Adsorption capacity of bare graphite was 41.18%, whereas that of oxidized graphite was 73.3%. The present results show that graphite oxide is a candidate material for the adsorption of Pb²⁺ ion from water.

Keywords: oxidized graphite; lead adsorption; XPS analysis; surface



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1. Introduction

The presence of heavy metals in waste water and industrial effluent is one of the major concerns for the environmental community and society at large [1]. Lead is one of the industrial pollutants entering the ecosystem through soil, air and water [2]. The main sources of lead from human activities include lead mining, smelting and the use of leaded gasoline lead-based paints and water pipes [3–6]. Lead (II) ion (Pb²⁺) is highly toxic at its exceeded level, responsible for intellectual disabilities in children and causes around 143,000 deaths annually in developing countries [7,8]. Furthermore, lead accumulates mainly in bones, brain, kidney and muscles, causing many serious disorders like anemia, kidney disease and nervous disorder [9,10]. The safe and efficient removal of lead is, thus, necessary to prevent it from causing harmful damage to the environment.

Removal of heavy metals from waste water has been achieved by using several methods such as adsorption, precipitation, electroplating, chemical coagulation, ion-exchange, membrane separation and electro kinetics [11–16]. Significant effort has been devoted to the removal heavy metals from waste water using adsorption as this method is an efficient and economical method [17,18]. A variety of sorbents, such as activated carbon [19], oxidized activated carbon [20], carbon nanotubes [21], oxidized carbon nanotubes [22], chemically modified carbon [23], synthetic oxide [24], oxide minerals [25] and agricultural based bio sorbents (stems, peels, husks, shells and leaves) [26–28], have been used extensively.

High surface area with active functional groups will lead to high active sites for the adsorption of Pb²⁺ ions. A variety of adsorbent materials containing different chemical functional groups, such as carbonyl, carboxylic, phenolic and aromatic groups, have been reported [29–32]. The presence of chemical functional groups such as carboxylic acid (-COOH) or alcohol (-OH) on the adsorbents will enhance the adsorption efficacy via dipole-dipole interaction such as cation π -bonding for Pb²⁺ adsorption [30,33]. The

mechanism for the adsorption of lead ions onto okra wastes was reported to be either via ion exchange or the formation of hydroxyl complexes [34]. Adsorption efficacy of Pb²⁺ ions not only depends on the structure and chemical properties of the adsorbent, but also on initial concentration of the metal ions, the pH of the solution, the dose of adsorbent and the temperature [35,36]. For example, Alghamdi et al. [36] have shown that adsorption capacity of Pb²⁺ ions on the polypyrrole-based activated carbon increases with increase in concentration and decreases with increase in adsorbent dose.

Oxidized graphite is a candidate material to remove large concentration of heavy metals as it has larger surface area decorated with organic functional groups, such as phenolic, carboxylic, carbonyl, quinoyl and lactonyl groups. As these functional groups are polar, the adsorption capacity of heavy metals is expected to be high. Furthermore, graphite powder is an inexpensive naturally occurring material that can be used in developing countries to purify water from heavy metals which cause chronic diseases.

In this study, we use standard electrochemical studies to determine the concentration of Pb^{2+} ions and XPS to confirm the adsorption of Pb^{2+} ions.

2. Materials and Methods

2.1. Reagents and Equipment

In order to prepare stock solution of Pb²⁺ ions (5 mM), lead nitrate (Aldrich, St. Louis, MO, USA) was used. The graphite powder from Aldrich was directly used and the particle size of the graphite powder was less than 20 μ m in diameter. A standard pH meter (PHS-W series microprocessor pH/mV meter, Shanghai, China) was used in all cases to measure the pH of the solutions. A computer controlled Metrohm Autolab potentiostat (Type of instrument PGSTAT 302N, Utrecht, The Netherlands) was used to record the electrochemical measurements. The three-electrode system in the autolab, consisted of a glassy carbon electrode (working electrode), a saturated silver-silver chloride (reference electrode) and a platinum electrode (counter electrode). For stirring solutions, a standard magnetic stirrer (IKA C-MAG HS7) was used. Using Omicron ESCA Probe spectrometer XPS analysis was carried out at Indian Institute of Technology, Madras, India. The Al K α band (h ν = 1486.7 eV) was used to determine the X-ray radiation. OriginPro 8 was used to analyze the resulting spectra.

2.2. Oxidation of Graphite

Graphite particles (<20 μ m in diameter) were oxidized by stirring 1 g of graphite powder with 20 mL of acid mixture prepared by mixing of 15 M HNO₃ and 18 M H₂SO₄ with 3:1 ratio for 12 h. Using distilled water the oxidized graphite powder was washed thoroughly until a neutral filtrate was obtained. Finally, the oxidized graphite powder was allowed to dry using a suction pump and kept in a desiccator.

2.3. Linear Range Determination of Square Wave Anodic Stripping Voltammetry (SWASV)

In order to determine the linear range for the determination of Pb^{2+} , a blank solution of pH 4.5 buffer (sodium acetate buffer solution of pH 4.5 was prepared by mixing 0.5 M sodium acetate and 0.5 M acetic acid) was used in the standard addition method. A 25.0 mL of the above buffer solution was taken in the cell and a glassy carbon electrode was dipped into the solution at a deposition potential of -1.1 V vs. saturated silver-silver chloride electrode and held for 120 s with stirring. Then the resulting solution was allowed for 10 s to reach equilibrium. Before each voltammogram potential was swept from -1.1 to -0.3 V at a scan rate of 0.1Vs⁻¹ a potentials of +0.8 V was applied for 180 s as conditioning potential to remove the remaining deposits on the surface in the previous experiment (see Supplementary Materials). A linear detection range of 1-14 μ M was produced by SWASV. Figure 1 shows the current versus potential graph of SWASV with standard addition of Pb²⁺ on glassy carbon electrode in a cell containing 25.0 mL acetate buffer solution (pH = 4.5) after a pre-concentration period of 120 s at -1.1 V and 10 s for equilibrium time. Figure 2 shows the standard addition plot of peak area from Figure 1 against added Pb²⁺ concentration (1-14 μ M).



Figure 1. The current versus potential graph of Square Wave Anodic Stripping Voltammetry (SWASV) with standard addition of Pb²⁺.



Figure 2. Standard addition plot of peak area from Figure 1 against added Pb^{2+} ion concentration (1–14 μ M).

Optimum initial concentration was determined by stirring 100 mg of oxidized graphite with 10.0 mL of different concentration (50–500 μ M) of Pb²⁺ solution for 30 min and the rest for 6 h. Using a suction pump mixtures were filtered and the standard addition method was used to determine the remaining Pb²⁺ ions in the filtrates.

2.4. Optimum Dosage of Oxidized Graphite for the Adsorption of Lead Ions (Pb²⁺)

Optimum dosage of oxidized graphite was determined by stirring 10.0 mL of 100 μ M of Pb²⁺ solution with 100 mg, 200 mg, 300 mg and 400 mg of oxidized graphite for 30 min. Then the resultant solutions were then equilibrated for 6 h. Finally, filtration was carried out on the mixtures using a suction pump to determine the remaining Pb²⁺ ions in the filtrates. The difference between the initial and final amounts of Pb²⁺ ions is the amount of Pb2+ adsorbed on the oxidized graphite.

2.5. Adsorption of Pb^{2+} Ions

The following equation was used to determine the percentage of lead adsorption from aqueous solution.

Percentage of adsorbed Pb²⁺ ions =
$$\frac{C_i - C_f}{C_i} \times 100\%$$
 (1)

where C_i and C_f are the initial and final concentrations of Pb²⁺, respectively.

The following equation was used to determine the adsorbed moles of Pb²⁺ ions.

Adsorbed moles of
$$Pb^{2+}$$
 ions = $\frac{C_i - C_f}{W} \times V \mod mg^{-1}$ (2)

where C_i and C_f are the initial and final concentration of Pb^{2+} ion, respectively, W (mg) is the amount of oxidized graphite powder and V (ml) is the volume of Pb^{2+} ion solution.

3. Results and Discussion

3.1. XPS Characterization

Samples of blank and oxidized graphite powders were characterized using XPS in order to calculate the amount of oxygen adsorbed on the surface of graphite. The XPS analysis was also carried out on the graphite and the oxidized graphite powders after the exposure of Pb²⁺ solution. Small portions of the samples were analyzed separately with the aid of XPS spectrometer and scans were carried out in the range between 0 and 1200 eV (see Figure 3).



Figure 3. XPS spectra of (a) blank graphite and (b) oxidized graphite in the range between 0 and 1200 eV.

Two significant peaks are observed with binding energies of 284.6 eV and 532.6 eV associated with the photo electrons emitted from C 1s and O1s levels, respectively (see Figure 3a). The XPS of oxidized graphite powder is shown in Figure 3b. The peaks correspond to the binding energies of 282.9 eV and 532.4 eV can be attributed to the photo electrons emitted from C 1s and O1s levels, respectively.

A quantitative analysis revealed that atomic percentages of O (O1s peak) and C (C1s peak) on the for blank graphite powder were 6.13% and 93.87%, respectively. The same analysis was also carried out on the oxidized graphite powder. The result shows that the atomic percentages of the peaks of O1s and C1s were 15.97% and 84.03%, respectively. The enhancement in the percentage of atomic oxygen from 6.13% to 15.97% confirmed the oxidation of blank graphite.

Next, XPS analysis was carried out at 30 °C on the sample of oxidized graphite powder after exposing of 100 μ M Pb²⁺ ion solution for a period of 6.5 h (stirring: 30 min; equilibrium: 6 h) and the pH of the solution was maintained at 6.5. The XPS spectra of oxidized graphite powder after exposure of Pb²⁺, with wide scan ranges of 0–1200 eV and 130–155 eV, are shown in Figure 4.



Figure 4. XPS spectra of oxidized graphite powder after exposure to Pb^{2+} with the scan range of (**a**) 0–1200 eV and (**b**) 130–155 eV.

The spectrum of oxidized graphite shows that there are two peaks at 138 eV and 143.8 eV in addition to C1s and O1s peaks (see Figure 4b). These peaks correspond to the emissions from Pb4f7/2 and Pb4f5/2, respectively, confirming the adsorption of Pb^{2+} .

3.2. Effective Concentration of Pb^{2+} Ions

The effective concentration of Pb²⁺ ions was examined by considering different concentration of Pb²⁺ with a fixed amount of oxidized graphite powder. A series of Pb²⁺ solution with different concentration (50–500 μ M) were prepared by diluting 5 mM of Pb²⁺ (stock solution). A total of 100.0 mg of oxidized graphite powder was then added to 10.0 mL of each solution and the mixture was stirred for 30 min and allowed to rest for 6 h. Then the concentration of Pb²⁺ ions in the remaining solution was determined using SWASV. Table 1 shows average adsorbed moles of Pb²⁺ on the oxidized graphite (100 mg) with initial concentration of Pb²⁺ from 50 μ M to 500 μ M.

Table 1. Average adsorbed moles of Pb^{2+} on the oxidized graphite (100 mg) with initial concentration of Pb^{2+} in the range between 50 μ M and 500 μ M.

Initial Concentration of Pb ²⁺ Ion (Ci μM)	Average Adsorbed Moles of Pb ²⁺ Ions in mol $mg^{-1}~(\times 10^{-10})$
50	44.863
100	73.333
200	80.806
300	82.313
400	85.816
500	84.400

As the initial concentration of Pb^{2+} increases from 50 μ M to 400 μ M, there is a gradual increase in the adsorption. The maximum adsorption of lead ions takes place at 400 μ M concentration and then at 500 μ M there is a very small decrease in the moles of adsorption of Pb^{2+} . It may be due to ion-ion repulsion. Once the active sites of oxidised graphite are saturated, there is a poor chance of further adsorption of Pb^{2+} and the strength of ion–ion repulsion will be dominant at high concentration of Pb^{2+} .

3.3. Effective Amount of Oxidized Graphite Powder on Adsorption

The effective amount of oxidized graphite for the adsorption of Pb^{2+} ion was examined by adding 100.0, 200.0, 300.0 and 400.0 mg of oxidized graphite powder individually to 10.0 mL of 100 μ M of Pb^{2+} ion solution. The concentration of remaining Pb^{2+} ions in the solution was determined using SWASV. Table 2 shows average moles of Pb^{2+} adsorbed on the oxidized graphite.

Table 2. Average adsorbed moles of Pb^{2+} ion (initial concentration of 100 μ M) versus dosage of oxidized graphite (between 100.0 mg and 400.0 mg).

Dosage of Oxidised Graphite (mg)	Average Adsorbed Moles of Pb ²⁺ Ions in mol $\rm mg^{-1}~(\times 10^{-10})$
100	73.333
200	81.613
300	80.883
400	81.410

The adsorption of Pb²⁺ increases with the increase of oxidized graphite powder from 100 mg to 200 mg. Adsorption decreases once the dosage of oxidized graphite exceeds 200 mg. The optimum dosage of oxidized graphite is 200.0 mg.

4. Conclusions

Using X-ray photoelectron spectroscopy (XPS) and standard electrochemical measurements, we examined the adsorption efficacy of Pb^{2+} ion on the surfaces of bare and oxidized graphite. The XPS measurement confirmed the oxidation of graphite with the final composite having 15.97% of oxygen and 84.03% of carbon, in comparison with the pristine graphite powder consisting of 6.13% oxygen and 93.87% carbon. The peaks, determined at 138 eV and 143.8 eV, associated with the emissions from Pb4f7/2 and Pb4f5/2, respectively, confirmed the adsorption of Pb²⁺ ions. The adsorption capacity of bare graphite was 41.18%, whereas that of oxidized graphite was 73.3%. This experimental study shows that oxidized graphite is a candidate material for the safe and effective capture of Pb²⁺ ions from waste water.

Supplementary Materials: The following are available online at https://www.mdpi.com/2311-562 9/7/1/23/s1, Figure S1: Potential versus current graph for standard addition of Pb²⁺ for graphite powder, Figure S2: Standard addition plot of peak area from Figure S1 against added Pb²⁺ concentration (1–5 μ M), Figure S3: Potential (V) versus current (A) graph for standard addition of Pb²⁺ solution for oxidized graphite powder, Figure S4: Standard addition plot of peak area from Figure S3 against added Pb²⁺ concentration (1–5 μ M).

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