



Evaluation of Pomegranate Peel as Bioadsorbent for Removing Lead Ions from Aqueous Solutions Using Phytoadsorption Technique

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ABSTRACT

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The removal of toxic heavy metals from ecosystems is a challenge. Looking for green solutions is always required. Phytoremediation is one of the most powerful, efficient, eco-friendly and sustainable methods. Moreover, phytoadsorption approach emerged in the last two decades as a potential, cheap, and benign method for the clean up of contaminated aquatic systems from heavy metals. This study investigates using the phytoadsorption technique for the pomegranate peels as biosorbent to remove lead ions from their aqueous solutions.. Different amounts of the dry biomass (5.0, 2.0, and 0.5 g) at varied shaking rates of 400, 600, 800 OSC/min and three periods of contact time were applied. The results of all experiments revealed that the dry pomegranate peels were able to remove up 91.1% of Pb²⁺ ions from their 1000-ppm aqueous solutions at room temperature and neutral pH. In addition, the pomegranate peel as a bioadsorbent has showed an adsorption capacity up to 96.1 mg/g. Therefore, the pomegranate peels can be used as potential bioadsorbents to remove lead ions from the contaminated aquatic systems.

1. INTRODUCTION

Anthropogenic activities and natural sources are responsible of environmental pollution. Heavy metals constitute the major contaminants of ecosystems; land, water, and air. While some heavy metals are essential elements for our life such as iron (Fe), copper (Cu), zinc (Zn), nickel (Ni) and manganese (Mn), the non-essential heavy metals like arsenic (As), lead (Pb), cadmium (Cd), mercury (Hg), chromium (Cr), and cobalt (Co) are very toxic elements and cause many diseases and disorders. Lead heavy metal, which classified in the second order

in toxicity behind arsenic metal, is a very toxic element and can cause damage to many soft tissues depending on the dosage, duration and type of exposure, bioaccumulation, and speciation of metal (Clarkson, 1987; Pandey and Madhuri, 2014; Caito & Aschner, 2015; Zaynab et al., 2022; Mitra et al., 2022). Bioremediation process is one of the effective techniques used for removing of toxic heavy metals and other pollutants from contaminated ecosystems. It can be divided into three main categories; phytoremediation

(plant-based bioremediation), zooremediation (animal-based bioremediation), and microbial remediation (microorganism-based bioremediation).

Phytoremediation process is a cheap, green, sustainable, and efficient approach to up-take toxic heavy metals from soil and aquatic systems (Prasad & Freitas, 2000; Rajakaruna et al., 2006; Sumiahadi & Acar, 2018, Yan et al., 2020; Bhat et al., 2022). This technology is described by different mechanisms depending on the type of plant biomass. In the living cell biomass, the phytoremediation mechanisms are phytoextraction, phytostabilization, phytodegradation, phytovolatilization, phytostimulation, and phytofiltration (Tangahu et al., 2011; Suman et al., 2018; Al-Khazan and Al-Zlabani, 2019; Nedjimi, 2021; Sabreena et al., 2022; Sharma et al., 2023). For the dead cell biomass, the phytoremediation is called phytosorption process, which classified into two main mechanisms; phytoadsorption and phytoabsorption. The phytoadsorption process is known as a biological physico-chemical interaction between the dead cell material of plant and pollutants for removing them from contaminated environment through physical or chemical adsorption. Phytoadsorption of heavy metals from their solutions is achieved by adsorption of metal ions onto the surface of plant particles (Etoriki et al., 2014; Al Saharty, 2014; Dubey et al., 2014; Karman et al., 2015; Redha, 2020; Kumar et al., 2021). There are many factors can control the efficiency of phytoadsorption of heavy metals using plant as biosorbent. These factors are initial metal concentration, plant material dosage, temperature, contact time, pH, presence of another cations, and chemical modifications of biosorbents (Redha, 2020). There is a wide spectrum of families in plant kingdom can be used in the phytoremediation techniques for the removal of heavy metals from contaminated systems (Prasad & Freitas, 2000; Sekhar et al., 2003; Srivastava et al., 2005; McGrath et al., 2006;

Zhi-xin et al., 2007; Probst et al., 2009; Dubey et al., 2014; Wao et al., 2014; Dube & Chingoma, 2016; Tang et al., 2019; Huynh et al., 2021; Ramadan & Balah, 2022). For instance, pomegranate peels were investigated for the phytoremediation of some heavy metals such as lead, copper, chromium, nickel, zinc and cadmium from aqueous solutions. They were able to remove high percentages of these metals and have showed a remarkable adsorption capacity (El-Ashtoukhy et al., 2008; Shartooh, et al. 2013; Ben-Ali, et al. 2017; Rashed, et al. 2020). More recently, our group has reported a simple, low-cost, green, and efficient phytoadsorption method for removing of lead ions from their aqueous solutions (Sharif et al., 2023 and 2023; Sharif et al., 2024). In this report, we investigated the ability of pomegranate peel as an absorbent for the removal of lead ions from an aquatic system using the phytoadsorption methodology.

2. MATERIALS AND METHODS

2.1 Sample Preparation

2.1.1 Lead Ion Solution (Pb^{2+})

Lead metal ion solution (1000 ppm) was prepared by dissolving lead nitrate, $Pb(NO_3)_2$, in distilled water which was utilized for the designed experiments.

2.1.2 Biomass

Fresh pomegranate fruit was collected from the local market, Benghazi city, Libya. The fruit peels were removed and then washed with water. After that, they were dried in dark place for 3 months and then they were ground into a powder form and stored in dark and dry place for the following steps.

2.2 Phytoadsorption Experiments and Analysis of Samples

All experiment procedures and samples' analysis were carried out according to the standard methods (Sharif et

al., 2024, 2023 & 2023, Etoriki et al., 2014). Specific amounts of dead plant biomass (5.0, 2.0, or 0.50 g) were added to a 100 mL of lead solution in a 500-mL container (polyethylene bottle), separately. Then, they were shaken well using an Instrumental Shaker (Flask Shaker SF1) at varied rates of 400, 600 and 800 OSC/min for three periods of time (30, 60 and 120 minutes). All experiments were carried out at room temperature and a neutral pH. All the lead solution-pomegranate peels mixture bottles were left for 24 hours to allow the solid matter to precipitate. Next, the peel solution mixtures were filtered using Whatman filter papers No 1. Filtrates were then diluted to the tithe (10%) of the original concentration (about ≤ 10 ppm) and acidified with nitric acid (0.5 mL of 60% HNO₃) at a roughly pH of 3 for the analysis step, and stored in freezer. Concentrations of the remained lead metal ions in all filtrates were detected using a flame atomic absorption spectroscopic (FAAS) instrument (Model: Perkin Elemer 500) at room temperature of 24 °C and pH of solutions at 2.4–3.8. Finally, removal percentage (%) of lead ions and adsorption capacity (mg/g) were calculated. The adsorption capacity q_e (mg/g) of each sample after equilibrium was calculated by mass balance relationship equation (El-Ashtoukhy et al., 2008; Ben-Ali, et al. 2017) as follows

$$q_e = (C_i - C_d) V/W$$

Where C_i is the initial concentration of lead ion solution (mg/L), C_d is the detected concentration of filtrate solutions (mg/L), V is the volume of the solution (L) and W is the mass of adsorbate (g).

3. RESULTS AND DISCUSSION

All samples showed a decrease of lead ion concentrations after shaking with amount of

pomegranate peels powder. These observations resulted from the adsorption effect of biomaterial via their surface negative charges of some functional groups of the biomolecules towards the cations of lead metal. Amounts of removed lead ions were affected by the loaded amount of pomegranate peel powder, the initial concentration of metal, and the contact time.

3.1 Biomass Amount: 5.0 g

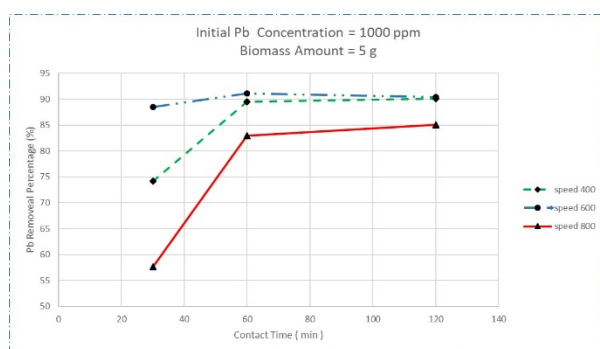
By using 5.0 g of loading pomegranate peels, the contact time (min) and shaking speed (OSC/min) of the 1000-ppm concentration of Pb²⁺ ions were investigated.

3.1.1 Effect of Contact Time at Different Shaking Speeds

Form the collected data, it was found that there was an increase of the removal percentage (%) of lead ions within the increased time (See Table 1, Figure 1). For example, at a shaking rate of 400 OSC/min, the percentage was 74.2% after 30 minutes of shaking (with adsorption capacity of 14.8 mg/g) followed by gradual increase to record 90.1% (with adsorption capacity of 18 mg/g) in 120 min (entries 1–3). At a shaking rate of 600 OSC/min, the situation slightly enhanced to reach the optimum removal percentage of 91.1% (with adsorption capacity of 18.2 mg/g) after 60 minutes (entry 5). However, at a shaking speed of 800 OSC/min, the lowest removal percentage was detected at 57.6% (with adsorption capacity of 11.5 mg/g) after 30 min (entry 7). Then, the adsorption of pomegranate peels increased to reach 85.1% of removal after 120 min (with adsorption capacity of 17 mg/g) under the same conditions (entry 9).

Table 1: Removal percentage of Pb²⁺ (%) from a 1000-ppm of lead solution and adsorption capacity (q_e) using 5.0 g of dry pomegranate peels

Entry	Shaking Speed (OSC/min)	Contact Time (min)	Pb Detected (ppm)	Pb Remained (%)	Pb Removal (%)	Adsorption Capacity (mg/g)
1	400	30	258	25.8	74.2	14.8
2	400	60	105.2	10.5	89.5	17.9
3	400	120	99.4	9.9	90.1	18.0
4	600	30	115.2	11.5	88.5	17.7
5	600	60	89	8.9	91.1	18.2
6	600	120	96	9.6	90.4	18.1
7	800	30	423.8	42.4	57.6	11.5
8	800	60	171	17.1	82.9	16.6
9	800	120	149.4	14.9	85.1	17.0

**Figure 1:** Effect of contact time and shaking rate on the removal percentage of Pb²⁺ using 5.0 g of dry pomegranate peels.

3.2 Biomass Amount: 2.0 g

Similarly, by using 2.0 g of loading pomegranate peels, the contact time (min) and shaking speed (OSC/min) of the 1000-ppm concentration of Pb²⁺ ions were investigated.

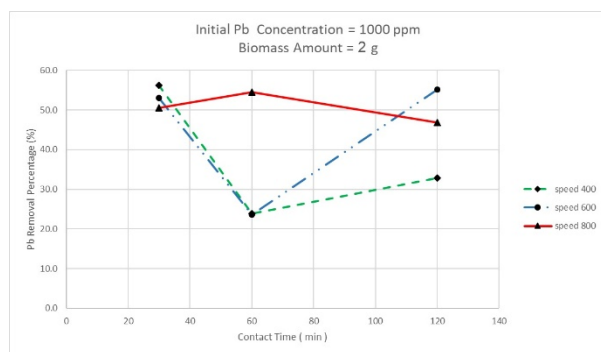
3.2.1 Effect of Contact Time at Different Shaking Speeds

Under these conditions, the situation was different. It was found that there was a fluctuation in the removal percentages (%) of lead ions within the increased time (See Table 2, Figure 2). For example, at a shaking rate of 400 OSC/min, the percentage was 56.2% after 30 minutes of shaking (with adsorption capacity of 28.1

mg/g) which represented the highest removal percentage in this group (entry 10). Then, a sharp decrease was observed in the removal rate of 23.9% (with adsorption capacity of 12 mg/g) in 60 min followed by an increase at 32.8% (with adsorption capacity of 16.4 mg/g) after 120 minutes of shaking (entries 11 and 12). With a speed of shaking at 600 OSC/min, the situation was similar. For instance, the Pb removal percentage was 53.1% (with adsorption capacity of 26.6 mg/g) after 30 minutes (entry 13). After that, it was followed by a sharp decrease at 23.6% (with adsorption capacity of 11.8 mg/g) after 60 min and then the removal percentage started to rise to 55.6% (with adsorption capacity of 27.6 mg/g) after 120 min (entries 14 and 15). In contrast, at a shaking rate of 800 OSC/min the first 30 minutes showed a lead removal percentage of 50.6% (with adsorption capacity of 25.3 mg/g) followed by an increase of 54.5% (with adsorption capacity of 27.3 mg/g) after 60 min (entries 16 and 17). After that, the percentage decreased to 46.9% (with adsorption capacity of 23.5 mg/g) during 120 minutes of shaking (entry 18).

Table 2: Removal percentage of Pb²⁺ (%) from a 1000-ppm of lead solution and adsorption capacity (q_e) using 2.0 g of dry pomegranate peels

Entry	Shaking Speed (OSC/min)	Contact Time (min)	Pb Detected (ppm)	Pb Remained (%)	Pb Removal (%)	Adsorption Capacity (mg/g)
10	400	30	438	43.8	56.2	28.1
11	400	60	761.3	76.1	23.9	12.0
12	400	120	671.6	67.2	32.8	16.4
13	600	30	469	46.9	53.1	26.6
14	600	60	763.5	76.4	23.6	11.8
15	600	120	448	44.8	55.2	27.6
16	800	30	494	49.4	50.6	25.3
17	800	60	455	45.5	54.5	27.3
18	800	120	531.2	53.1	46.9	23.5

**Figure 2:** Effect of contact time and shaking rate on the removal percentage of Pb²⁺ using 2.0 g of dry pomegranate peels.

3.3 Biomass Amount: 0.5 g

By uploading 0.5 g of loading pomegranate peels, the contact time (min) and shaking speed (OSC/min) of the 1000-ppm concentration of Pb²⁺ ions were also investigated.

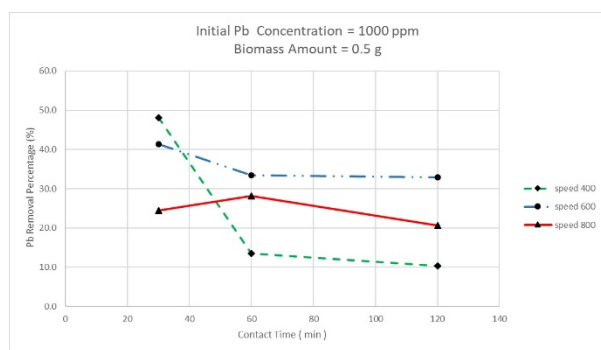
3.3.1 Effect of Contact Time at Different Shaking Speeds

In general, , under the three different speeds of shaking, there was a decrease in the removal percentages of Pb²⁺ during the applied contact times (see Table 3, Figure 3). For example, at a shaking rate of 400 OSC/min, the removal percentage was 48.1% after 30 minutes of

shaking (with adsorption capacity of 96.1 mg/g) which represented the highest removal percentage in this group (entry 19). Then, a significant decrease of removal percentage was observed to record 13.5% (with adsorption capacity of 27 mg/g) after 60 min (entry 20), followed by a 10.3% (with adsorption capacity of 20.6 mg/g) after 120 minutes of shaking, which represented the minimum adsorption capacity (entry 21). Similarly, the removal percentages of lead ions at a shaking rate of 600 OSC/min showed a high adsorption capacity after the first 30 minutes by 41.3% (with adsorption capacity of 82.6 mg/g) (entry 22). Next, after 60 and 120 minutes of shaking the removal percentages of lead ions were 33.4 and 32.9% (with adsorption capacity of 66.8 and 65.8 mg/g), respectively (entries 23 and 24). On the other hand, the removal percentage pattern was similar to the two previous cases (at shaking rates of 400 and 600 OSC/min) except for the first 30 minutes of shaking which recorded 24.5% (with adsorption capacity of 49 mg/g) (entry 25). The removal percentage in the next experiment (entry 26) increased to 28.2% (with adsorption capacity of 56.4 mg/g) after 60 minutes followed by a decrease at 20.6% (with adsorption capacity of 41.2 mg/g) after 120 min (entry 27).

Table 3: Removal percentage of Pb²⁺ (%) from a 1000-ppm of lead solution and adsorption capacity (q_e) using 0.5 g of dry pomegranate peels.

Entry	Shaking Speed (OSC/min)	Contact Time (min)	Pb Detected (ppm)	Pb Remained (%)	Pb Removed (%)	Adsorption Capacity (mg/g)
19	400	30	519.5	51.9	48.1	96.1
20	400	60	865	86.5	13.5	27.0
21	400	120	897	89.7	10.3	20.6
22	600	30	587.3	58.7	41.3	82.6
23	600	60	666.2	66.6	33.4	66.8
24	600	120	671.2	67.1	32.9	65.8
25	800	30	755.3	75.5	24.5	49.0
26	800	60	717.7	71.8	28.2	56.4
27	800	120	794	79.4	20.6	41.2

**Figure 3:** Effect of contact time and shaking rate on the removal percentage of Pb²⁺ using 0.5 g of dry pomegranate peels.

3.4 Effect of Biomass Amount on Adsorption Capacity:

The adsorption capacity, q_e , of dry pomegranate peels for removing lead ions from their aqueous solutions significantly increased by decreasing the loaded amounts of dry biomass for all applied shaking rates (see Table 4, Figure 4). In general, it was observed that the best adsorption capacity obtained from using the lowest amount of biomass and the slower shaking rates at the same contact time. For example, the highest adsorption capacity using 0.5 g of dry pomegranate peels was 96.1 mg/g after 30 min of shaking at 400 OSC/min (entry 3). However, the lowest adsorption capacity of the dry material (5 g) was 11.5 mg/g at 800 OSC/min of shaking at the same contact time (entry 7).

Table 4: Adsorption capacity (mg/g) of dry pomegranate peels (g) after shaking rate (OSC/min) at contact time (30 min).

Entry	Shaking Speed (OSC/min)	Biomass Amount (g)	Pb Detected (ppm)	Pb Remained (%)	Pb Removal (%)	Adsorption Capacity (mg/g)
1	400	5.0	258	25.8	74.2	14.8
10	400	2.0	438	43.8	56.2	28.1
19	400	0.5	519.5	51.9	48.1	96.1
4	600	5.0	115.2	11.5	88.5	17.7
13	600	2.0	469	46.9	53.1	26.6
22	600	0.5	587.3	58.7	41.3	82.6
7	800	5.0	423.8	42.4	57.6	11.5
16	800	2.0	494	49.4	50.6	25.3
25	800	0.5	755.3	75.5	24.5	49.0

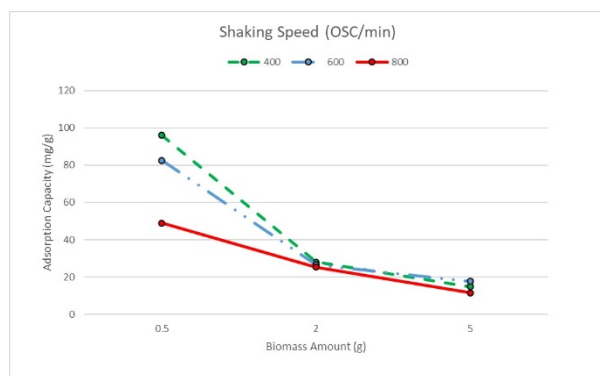


Figure 4: Effect of biomass amount on adsorption capacity of Pb²⁺.

1 CONCLUSION

Contamination of aquatic systems with heavy metals is a huge concern, globally, and it is a real threat to human health. Phytoremediation techniques, in particular phytoadsorption process, represent one of the greenest and efficient processes to remove heavy metals from polluted environment. The results of the current study revealed that the dry pomegranate peel biomass can be used as an excellent biosorbent for the removal of lead ions from their highly concentrated aqueous solutions with a remarkable adsorption capacity. Therefore, this developed approach can be applied for the cleaning up of contaminated aquatic environmental systems from heavy metals.

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Conflicts of Interest

The authors declare that there are no conflict of interest.

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Authors' Contributions

All authors contributed to the work and approved it for publication.

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