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A cost-effective and environmentally friendly approach for Pb (II) removal from water by using a novel plant-base adsorbent

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Abstract

The indiscriminate release of toxic heavy metals (HMs) such as lead (Pb) in to surface/ground water has led to several serious environmental problems. In this research, the potential of *Pomegranate* seed powder (PSP) was evaluated as a natural adsorbent for Pb removal from contaminated water. Real samples of contaminated water from Babol River were used to evaluate the performance of PSP. The effect of main operation parameters such as PSP dosage, contact time, initial concentration of HMs and pH on removal efficiency were studied. Moreover, SEM, EDX and FTIR analyses were performed to investigate HMs removal mechanism. According to the results, adding 1.5 g/L of PSP to samples could increase the removal efficiencies of Pb to 88% within 30 min contact time. Initial concentrations of Pb and pH variation showed negative effect on Pb removal. The results of SEM-EDX and FTIR analyses revealed that PSP has good feature which can effectively adsorb HMs through physico-chemical mechanisms. This study suggests HMs removal by PSP as a promising approach for heavy metals removal from contaminated surface water.

Key words: Water treatment; Natural adsorbent; Lead removal; Pomegranate seed powder.

1.Introduction

Clean water is vital for human being. It is generally obtained from rivers, lakes, and groundwater sources. Water pollution as the results of human activities becomes one of the serious ecological problem that we are facing with [1]. Heavy metals (HMs) are considered to be one of the most toxic water pollutants [2]. In general, HMs are released into the water bodies via industrial, agriculture and domestic waste, or even from acidic rain breaking down soils and liberating HMs into rivers, lakes and streams. [3]. While, some HMs such as Zn(II), Fe(III), Ni(II) are known as less toxic HMs, Cr(VI), Cd(II), As(V), Hg(II), Mn(II), Pb(II) are known as highly toxic HMs,. These chemicals will be absorbed by plants and have the possibility of being transferred to other living beings which consume those plants. In such a way, these harmful chemicals also affect human beings too [4].

HMs transport to human through air, water and food chains. The biomagnifications and bioaccumulation in the living organisms and human bodies can cause unwanted serious health complications, such as cancer, kidney and liver damage that may lead to death at very high exposure [3]. HM ions are highly toxic in nature even at low concentrations with high density [1]. Among HMs, Lead (Pb) are the most common ones derived from both natural and anthropogenic sources [5]. Pb can cause severe damage in nervous and genital system if it gets enrichment in the human body [6]. Therefore, exploring economical and renewable techniques removing above HMs from water is important for safe water production. In recent decades, many techniques have been introduced for treating contaminants in water, which include advance oxidation processes (AOPs), electrocoagulation, distillation, desalination, membrane filtration, reverse osmosis, electrodialysis, coagulation, filtration, ion exchange, precipitation and adsorption [7].

Among different HMs removal methods, the adsorption/chemical precipitation process is the cheapest and the most effective treatment technology adopted by many industries. To develop an innovative treatment process, the adsorption was found for removing or reducing the concentration of metal ions. This method has several benefits such as; the capital investment is low, removal of metal ions by selective basis and no sludge generation. The pollutants existing in the wastewater has been removed by using several organic and inorganic adsorbate materials [8]. Many organic waste materials were used to reduce the concentration of toxic pollutants from aqueous medium. Among the various organic waste materials, Pomegranate seed powder (PSP) are the most important material which is produced from pomegranate fruit (*Punica granatum* L.) has been suggested a sustainable natural adsorbent [9]. PSP which can be considered as a food-waste, is inedible, inexpensive material (almost free of charge) and available worldwide. PSP is composite of organic material which contains 2.3% of ash. Also, this consists of 12.5 % moisture, 13.5% total protein, 45.5% total carbohydrate and 15.2% total lipid. However, lead (Pb) as heavy metal is toxic in low concentrations to both plants and animals. Removing/reducing the concentrations of this heavy metal is necessary. To the best of our knowledge limited research have been conducted to investigate the potential of PSP and a natural adsorbent for HMs removal from water.

Therefore in this study, Pomegranate seed powder (PSP) was applied as a natural adsorbent for lead (Pb) removal from aqueous. The effect of main operation parameters such as PSP dosage, contact time, initial concentration of HMs and pH on removal efficiency were studied to identify the optimal conditions. Moreover, SEM, EDX and FTIR analyses were performed to investigate HMs removal mechanism.

2. Materials and methods

2.1. Polluted water sampling

One liter of river water samples were collected from five different points of Babol River, Mazandaran, Iran. The average concentrations of heavy metals in the river at different sampling points were presented in Table 1. According to the Table 1, samples from point 4 showed the highest level of heavy metals due to the industrialization in the urban area and contaminated water injection in to the river. In particular, the level of Pb was much higher than the accepted standard level for drinking ($Pb < 10 \mu\text{g/L}$) and agriculture ($Pb < 5\text{m g/L}$) purposes (EPA). pH level was adjusted by adding other materials including sodium chloride (NaCl) and hydrochloric acid (HCl) for pH adjustment of the water were purchased from Sigma-Aldrich in commercial grade.

Table 1. Heavy metals content of Babol River water at different sampling points

Code	Location	pH	T _w	Cu	Pb	Cr	Al	Zn
			(°C)	μg/L	μg/L	μg/L	μg/L	μg/L
S1	"E5.72'48"N 52° 13'14°	5.5	10°	2262.2	88.57	1.5	87.7	0.1
S2	"E49.301'41"N 52° 5.0434'2636°	6.0	11°	2362.9	292.2	2.6	145	0.5
S3	"E49.255'41"N 52° 5.3455'2636°	6.3	15°	2419.5	306.9	5.9	173	0.6
S4	"E30.485'38"N 52° 39.617'3636°	6.6	16.5°	2483.5	537.5	6.1	182	1.1
S5	"E44.097'38"N 52° 5.147'4236°	6.8	14°	2506.9	498.1	4.0	146	1.3
Standard (EPA)		7		1300	15	50	200	5000

2.2. Preparation of Pomegranate seed powder

The *Pomegranate* seeds (PS) were obtained from a local juice shop in Babol, Mazandaran, IRAN. Since the crushed seeds of *Pomegranate* were a type of food-waste produced in the process of *Pomegranate* juicing, they were provided free of charge. The PS were washed with distilled water to completely remove any impurities from the seeds. After that, the PS were oven dried in the oven at 40 °C for 24 h. To prepare *Pomegranate* seeds powder (PSP), the dried seeds were grinded and passed through a 70-mesh sieve. The PSP was then added directly to the sand filtration. Fig. 1 illustrates the PS and PSP used in this study.



Fig. 1. Pomegranate seed powder (PSP) used in this study

2.3. Batch adsorption studies

The HMs concentrations in the polluted water before and after treatment were measured using an atomic adsorption spectrometer (ICP-OES, USA). The removal efficiency (%) and adsorption capacity (mg/g) of HMs were calculated using Eq. (1) and Eq. (2). respectively:

$$\text{Removal efficiency } (R) = [(C_0 - C_e)/C_0] \times 100 \quad (1)$$

$$\text{Adsorption capacity } (q_e) = (C_0 - C_e)V/M \quad (2)$$

where C_0 and C_e (mg/L) are the initial and final concentrations of HMs in the water before and after treatment, respectively. M is the mass of the adsorbents (g) and V is the volume of the polluted water (L) which was equal to 0.25 L in this study.

2.4. Kinetic studies

The pseudo-first-order (PFO), pseudo-second-order (PSO) and interparticle diffusion (ID) kinetics models were used to investigate the adsorption rate of the Lead modified by PSP adsorbent in this study. The PFO, PSO and ID models are represented by Eq.s 3, 4 and 5, respectively [10]:

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

$$q_t = k_i t^{0.5} + C \quad (5)$$

where q_e and q_t (mg/g) are the adsorption capacity at equilibrium and t (min), k_1 (1/min), k_2 (g/mg.min) and k_i (mg/g.min^{-0.5}) are the rate constants of PFO, PSO and ID, respectively, and C is denoted the constant of boundary layer thickness.

2.5. Adsorption isotherm studies

The adsorption isotherm allows to identify the mechanism of the contaminant adsorption by representing the relationship between the adsorbate concentration (Pb) and the adsorbent (PSP) capacity (q_e) at equilibrium [11]. Several isotherm models are presented evaluate the adsorption process. In this study, Langmuir and Freundlich isotherm models were used as follows [10]:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (6)$$

$$q_e = K_f C_e^{1/n} \quad (7)$$

where q_m and q_e are the maximum adsorption capacity and the capacity at the equilibrium condition (mg/g), respectively. C_e is the equilibrium concentration of the HMs ions (mg/L) and K_L is the Langmuir isotherm constant (mg/L) which indicates how strongly an adsorbate molecule is attracted onto a surface. K_f and n are Freundlich constants, which represent the adsorption capacity of the adsorbent and the heterogeneity of the system, respectively.

2.6. Characterization details.

SEM image and EDX detector was applied to investigate the surface morphology on the PSP by using SNE-4500 M, SEC Co., Tokyo, Japan. Functional groups of the adsorbent were analyzed using a FTIR spectrometer (Bruker Tensor 27 FT-IR, Germany). A microprocessor pH meter (HANNA pH211, USA) was used to measure samples pH. All the measurements were performed three times and the results were reported as the mean values \pm standard deviation (SD).

3. Results and discussion

3.1. Batch adsorption studies

Fig. 2 shows the effects of PSP dose, initial Pb concentration, initial pH and contact time on the Pb removal performance. The results obtained from the batch studies will be helpful for finding out the optimum level of various adsorption parameters for obtaining the maximum removal efficiency of heavy metal ions.

Fig. 2a show the effect of adsorbent dosage of PSP on the removal efficiency of Pb at particular contact time of 60 min. The maximum removal efficiency of 88% for Pb was achieved by adding 1.5 g/L PSP. The initial concentration of the HMs affects the adsorption rate of the adsorbents [5].

Fig. 2b illustrate the effect of various initial concentrations of Pb (from 0.5 to 50 mg/L) on the final removal efficiency of the HMs by the PSP. The other parameters such as adsorbent dosage and contact time were kept constant. It was observed that by increasing the initial concentration of HMs up to 10 mg/L, the removal efficiency of Pb increased from 88% to 94%, The initial pH of the water directly affects the HMs adsorption process [12].

Fig. 2c represent the effect of pH variation (from 3 to 11) on the removal efficiency and final concentrations of Pb after 60 min of treatment with 1.5 g/L of PSP. This adsorbent showed the best performance at pH around 7 which the initial pH of the river water was 6.6. The observation of maximum adsorption under natural pH condition ($6 < \text{pH} < 7$) has been previously reported by Kumar et al., Mandal et al., Priya et al. and Shi et al. when they used Aminoalkyl-organo-silane, activated carbon, rice husk ash and sodium alginate, respectively as adsorbent for HMs removal from wastewater [5], [11]–[13].

Fig. 2d show the removal efficiency of Pb as a function of contact time under different dosages of PSP adsorbent. According to the result, contact time showed a direct effect on the HMs adsorption by the PSP. The HMs removal efficiency increased rapidly with an increase in contact time during the initial stages in all the cases. For example, when using 1.5 g/LPSP the removal efficiencies of Pb increased up to 88 % within 30 min contact time. This might be associated with the more availability of active binding sites on the PSP at the initial stage of filtration [13].

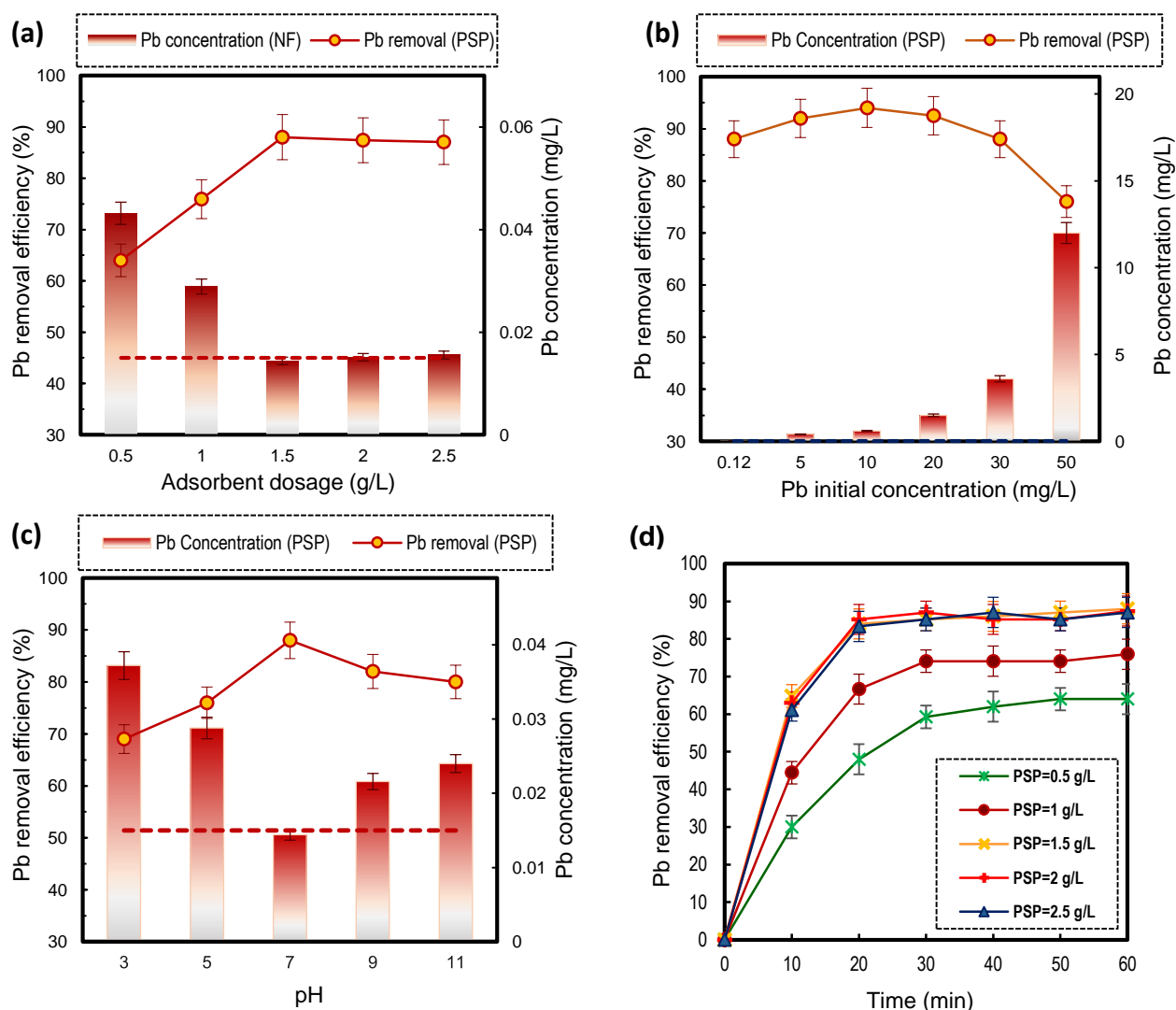


Fig. 2. Impact of adsorption factors of (a) dose level, (b) initial concentration of metal ion, (c) pH, (d) contact time on the removal efficiency of (Pb) by PSP

3.2. Kinetic studies on metal ION adsorption

Three kinetic models of pseudo-first-order (PFO) (Eq.3), pseudo-second-order (PSO) (Eq.4) and intraparticle diffusion (ID) (Eq.5) were used to analyze the rate of HMs sorption by the PSP during filtration process (Fig. 3). The initial concentrations of Pb^{2+} in the river ($Pb^{2+} = 0.12$ mg/L) were considered in the adsorption kinetic study. According to Fig. 3, the adsorption of HMs by the PSP increased rapidly during the initial stage and then became almost constant which indicates the majority of the adsorption ($> 90\%$) occurred within the first 10 min. After that, limited active surfaces were available for the HMs adsorption. The results revealed that in the case of Pb adsorption by the PSP, PSO model (PSP: $R^2 = 0.986$) fitted slightly better than PFO (PSP: $R^2 = 0.983$) and much better than ID (PSP: $R^2 = 0.823$).

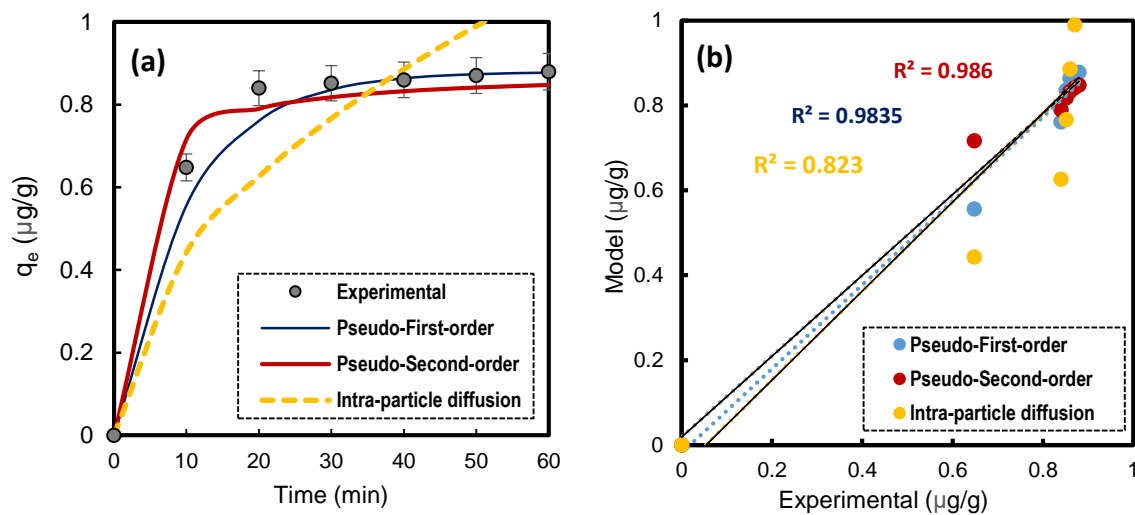


Fig. 3. Evaluation of the kinetics models for (a) Pb adsorption by PSP and (b) the models R^2

3.3. Adsorption isotherm studies

Fig. 4 show the result of adsorption isotherms of Pb for the adsorbent of PSP. Langmuir (Eq. 6) and Freundlich (Eq. 7) isotherms models were used to describe the isotherm adsorption. As can be seen from Fig. 4, the adsorption capacity of the PSP increased with increasing the initial concentration of HMs. In the case of PSP, the maximum adsorption capacity of 316.67 $\mu\text{g/g}$ were achieved for Pb ion at equilibrium concentrations of 12 mg/L. The Langmuir model showed a better match with the experimental data ($R^2 = 0.989$ and $q_{\text{max}} = 276.88$ $\mu\text{g/g}$ for Pb), compared to the Freundlich model ($R^2 = 0.969$ and $q_{\text{max}} = 324.04$ $\mu\text{g/g}$ for Pb) for PSP. Generally, it has been reported that the Langmuir model indicates monolayer adsorption [14]–[16]. As a result, the isotherm analysis revealed that HMs adsorption by PSP was mainly through monolayer adsorption.

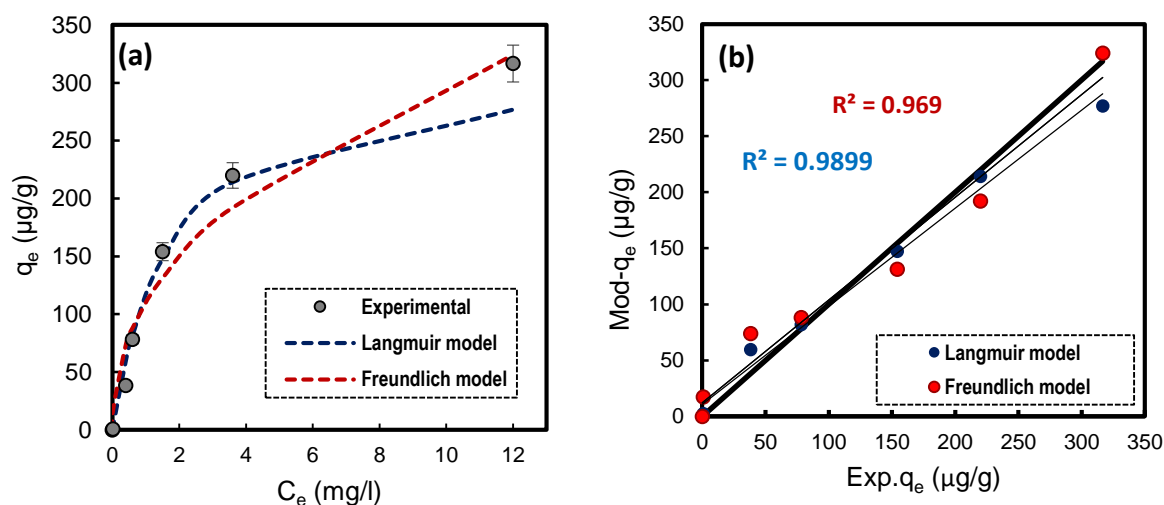


Fig. 4. Evaluation of the isotherm models for (a) Pb adsorption by PSP and (b) the models R^2

3.4. SEM & EDX analysis

Fig. 5 shows the surface morphology of the PSP adsorbent determined by SEM and EDX analyses. According to the SEM image (Fig. 5a) analysis with a magnification of 1000X, PSP showed a fibrous structure with a deep porous network structure on its surface. Fig. 5b shows the energy dispersive X-Ray (EDX) analysis of the PSP adsorbent. The weight percentages (w %) of other elements in PSP were observed as the following order: C (50.16 w %) > O (22.55 w %) > N (11.65 w %) > P (7.98 w %) > S (3.59 w %) > K (1.81 w %) > Ca (1.43 w %) > Mg (0.83 w %). However, according to the previous studies, the presence of some inorganic elements in the adsorbent improves the adsorption capacity of the adsorbent through electro attraction forces [17]. Ultimately, the result of EDX analyses prove that adsorbent has the potential of being used as appropriate adsorbent for heavy metals.

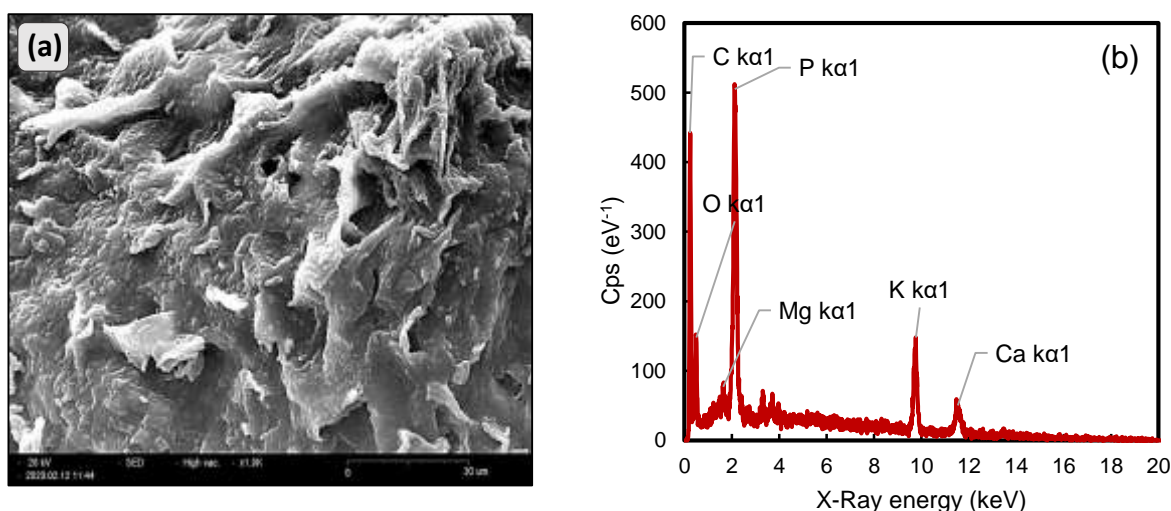


Fig. 5. (a) SEM images with a magnification of 1000X and (b) EDX analysis of PSP

3.5. FT – IR studies

Fig. 6 shows the FTIR analysis of the adsorbent of PSP. A strong peak at 3355.58 cm^{-1} was observed for PSP, corresponding the vibration stretching of water (O-H) and primary amine groups (N-H) [18]. Three peaks of 2999.25 , 2928.13 and 2855.72 cm^{-1} were observed within the wavenumber range of $2800 - 3000\text{ cm}^{-1}$, representing the $-CH$ stretching groups [19]. The observation of these three peaks for PSP indicates the presence of fatty acids and proteins in the seeds. A strong peak at 1743.30 were observed for PSP could be assigned to stretching vibration $C=O$ ester groups in carbonyl and carboxyl [20]. For the PSP, strong peaks were observed at 1576.79 and 1576.52 cm^{-1} which related to the aromatic $C=C$ group [21]. The presence of $-CH_2$ binding vibrations in the PSP was confirmed by the peaks at 1419.59 and 1426.43 cm^{-1} for PSS. These peaks suggest that the fine structure can be formed at the low energy region due to the hydrogen bonding [11]. The peaks at 1159.70 and 114.31 cm^{-1} for PSP was related to $C-O-C$ bonds' vibration [22]. The peaks which were observed between $800 - 1100\text{ cm}^{-1}$ for the PSP (938.41 , 989.07 and 1099.75 cm^{-1}) was related to $C-O$ stretching in alcohols and phenols [23]. All the other peaks bellow 800 cm^{-1} for PSP (619.69 , 651.10 , 725.21 and 760.88 cm^{-1}) was corresponded aromatic $C-H$ bending vibrations [20]. The FTIR analysis revealed that the PSP has active sites of metal ions adsorption.

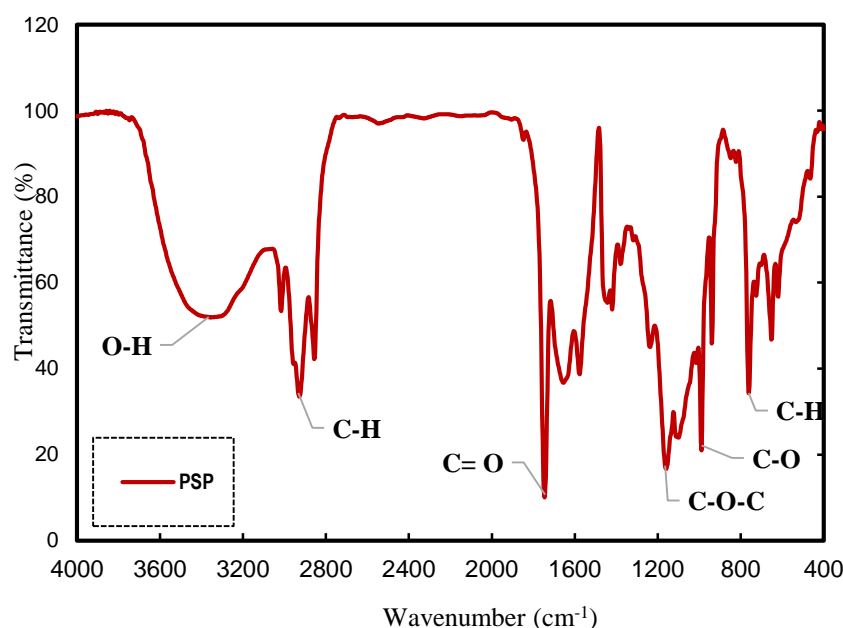


Fig. 6. FTIR spectra of the adsorbents of PSP

3.6. Comparison with previous

A comparison between the adsorbents suggested in this study and that in the literature is presented as Table 2. According to Table 2, using *pomegranate* seed powder (PSP) as Natural adsorbent could significantly improve HMs removal. Adding materials for HMs removal were also reported for improving removal. For example, Kumar et al. 2022 [16] added Aminoalkyl-organosilane (AOS) to sand filtration to improve Ar removal from wastewater.

Table 2. Comparison between the adsorbents suggested in this study and that in the literature

adsorbent	pollution	Removal	Ref.
Biochar	Gram-negative and Gram-positive bacteria	42.9%	[24]
Zero-valent iron	Bacterial capture and inactivation	99%	[25]
AOS	Arsenic	60%	[16]
Biochar	Pb ²⁺	66.5%	[26]
Rice husk ash	Cr ⁶⁺ , Pb ²⁺ and Zn ²⁺	87.12 %, 88.63 % and 99.28%	[1]
PSP	Pb ²⁺	88%	This study

4. Conclusion

This study suggests a novel, sustainable and environmentally friendly approach for removal of lead (Pb) from contaminate river water. The results revealed that the removal of Pb was pH dependent, and the optimum pH was determined as around 7. The highest percentage removal of Pb was found to be 88% for PSP (1.5 g/L) at 30 min of contact time. The experimental results proved that PSP could be employed successfully as a cost effective, benign, environmentally friendly adsorbent for the remediation of heavy metals in this environment.

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