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# Preparation and characterization of activated carbon from *Amygdalus Scoparia* shell by chemical activation and its application for removal of lead from aqueous solutions

**Research Article**

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**Abstract:** Two series of activated carbon have been prepared by chemical activation of *Amygdalus Scoparia* shell with phosphoric acid or zinc chloride for the removal of Pb(II) ions from aqueous solutions. Several methods were employed to characterize the active carbon produced. The surface area was calculated using the standard Brunauer-Emmet-Teller method. The microstructures of the resultant activated carbon were observed by scanning electron microscopy. The chemical composition of the surface resultant activated carbon was determined by Fourier transform infrared spectroscopy. In the batch tests, the effect of pH, initial concentration, and contact time on the adsorption were studied. The data were fitted with Langmuir and Freundlich equations to describe the equilibrium isotherms. The maximum adsorption capacity of Pb(II) on the resultant activated carbon was 36.63 mg $g^{-1}$ with $H_3PO_4$ and 28.74 mg $g^{-1}$ with $ZnCl_2$. To regenerate the spent adsorbents, desorption experiments were performed using 0.25 mol $L^{-1}$ HCl. Here we propose that the activated carbon produced from *Amygdalus Scoparia* shell is an alternative low-cost adsorbent for Pb(II) adsorption.

**Keywords:** *Chemical activation • Activated carbon • Lead • Amygdalus Scoparia shell • Agriculture waste*



## 1. Introduction

More than 800 unique organic and inorganic chemical compounds have been identified in drinking water. These compounds are derived from industrial and municipal discharge, urban and rural runoff, natural decomposition of vegetable and animal matter, and from water and waste water chlorination practices. Liquid effluents from industry also discharge varying amounts of a variety of chemicals into surface and ground water. Many of these chemicals are carcinogenic and cause many other ailments of varying intensity and character. Several methods such as coagulation, oxidation, aeration, ion exchange, and activated carbon adsorption have been used for the removal of these chemical compounds. Many studies including laboratory tests and field operations have indicated that adsorption onto activated carbon is perhaps the best broad spectrum control technology available at the present moment [1].

The steady increase in pollution necessitates the analysis and monitoring of toxic species that could become a serious potential hazard if not controlled [2]. Lead is one of the most toxic elements, has an accumulative effect and is an environmental priority pollutant [3]. The harmful effects on human health caused by lead contamination, are well-known. Among them, the reduction of enzymatic activity, kidney function and

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neuromuscular difficulties have been reported [4]. The U.S. Environmental Protection Agency (EPA) has classified lead as a group B2 (probable) human carcinogen [5]. Nowadays, there are legal restrictions concerning lead release into the environment. Nevertheless, it is used as a raw material in the manufacturing industry such as automotive batteries, ceramic and ink [4]. Furthermore, lead is a byproduct of several industrial processes in the production of fertilizers and pesticides. In both cases, it could either be delivered to the environment by inadequate manufacturing processes or by accident [5]. Environmental and health problems fundamentally arise from the use of gasoline antiknock products and paint pigments [6]. As a consequence, the World Health Organization (WHO) has established the maximum allowable limit of 10 ng $mL^{-1}$ for lead in drinking water. It is, therefore, important to monitor the lead level in environmental samples.

Activated carbon (AC) is known to be a very effective adsorbent due to its highly developed porosity, large surface area (that can reach 3000 $m^2$ $g^{-1}$), variable characteristics of surface chemistry, and high degree of surface reactivity [7,8]. These unique characteristics make activated carbon a very versatile material, which has been studied not only as an adsorbent, but also as a catalyst and catalyst support used for different purposes such as the removal of pollutants from gaseous or liquid phases and the purification or recovery of chemicals [9]. However, due to its high production cost, these material tends to be more expensive than other adsorbents. Currently, there are many studies on the development of low-cost adsorbents, namely the use of waste materials for that purpose. Also, several reviews report a great deal of work done on their application for the removal of specific pollutants from the aqueous phase, mainly heavy metals and dyes [10-14].

Up to date, many studies report that the removal of heavy metals by AC is economically favorable and technically easy [15,16]; therefore, AC is widely used to treat waters contaminated with heavy metals. Adsorption of metallic ions from aqueous solution is far from being a straightforward process. Metallic species are small in size, being frequently charged in solution; therefore, the predominant interactions in their adsorption process on AC are electrostatic [17]. The predominant factors that control the extent of adsorption on AC are [18]: (i) the chemistry of the metal ion (speciation) or metal ion complex; (ii) the solution pH and the point of zero charge of the surface; (iii) the surface area and porosity (narrow and wider microporosity); (iv) the surface composition (oxygen functionality); and (v) the size of the adsorbing species (hydrated ions in the range 1.0–1.8 nm).

In this study, *Amygdalus Scoparia* shell from Kerman province in Iran was used to prepare activated carbon as a new sorbent for the removal of Pb(II) from aqueous solution. To the best of our knowledge, this material was never used before for this application. The effects of initial adsorbate concentration, pH, contact time, and capacity of sorbent on the removal of Pb(II) were studied.

# 2. Experimental Procedure

## 2.1. Reagents and instrumentation

A stock solution of lead at a concentration of 1000.0 mg $L^{-1}$ was prepared by dissolving appropriate amounts of $Pb(NO_3)_2$ (Merck, Darmstadt, Germany) in $HNO_3$ (0.2 mol $L^{-1}$). Working reference solutions were prepared daily by stepwise dilutions from stock solution. All reagents were of analytical reagent grade. A SensAA GBC (Dandenong, Australia) atomic absorption spectrometer was used for measuring Pb(II) in air-acetylene flame. A Metrohm pH meter (Herisau, Switzerland) was employed for pH measurements. The concentrations of the lead solutions were obtained using data from a standard calibration curve.

## 2.2. Preparation of activated carbon

*Amygdalus Scoparia* shells (AS) were used as a precursor for preparation of activated carbon. AS shells were obtained from Kerman province in Iran. First, the AS shells were washed with distilled water to remove all foreign materials. Then, the shells were ground in a laboratory mill, dried at 110°C for 10 h, and sieved to a uniform particle size (40 – 60 mesh) prior to the activation process. This material was divided to two parts. One part was mixed with a concentrated phosphoric acid solution (weight ratio of 1:2) and allowed to soak for 24 h at room temperature. The excess $H_3PO_4$ solution was then decanted and the $H_3PO_4$-treated *Amygdalus Scoparia* shells (PAS) was then dried in an oven for 24 h at 100°C. The remaining part was mixed with a $ZnCl_2$ solution (weight ratio of 1:2) and allowed to soak for 24 h at room temperature. The excess $ZnCl_2$ solution was then decanted and the $ZnCl_2$ treated *Amygdalus Scoparia* shells (ZAS) were dried in an oven for 24 h at 110°C. The PAS and ZAS were then placed in a muffle furnace, heated at a rate of 10°C $min^{-1}$ to 550°C, and held at this temperature for 3 h. The resultant activated carbon was cooled to room temperature and was washed sequentially several times with hot water and 0.1 mol $L^{-1}$ hydrochloric acid to remove the residual $H_3PO_4$ and $ZnCl_2$, respectively. The resultant activated carbon was then washed sequentially several times with distilled water. Wash waters were tested with lead

**Figure 1.** Photograph of the raw material

nitrate and silver nitrate solutions for the detection of phosphate and chloride ions, respectively. Following washing, the activated carbon was dried at 110°C and sieved to obtain the desired particle size (0.300-0.425 mm). The activated carbon prepared after the $H_3PO_4$ and $ZnCl_2$ treatment will be indicated as PASAC and ZASAC, respectively.

### 2.3. Characterization of the raw *Amygdalus Scoparia* shells and prepared adsorbents

A photograph of the raw material is given in Fig. 1. The chemical composition of the raw material is: carbon 57.8%, hydrogen 5.4%, nitrogen 2.8, oxygen 33.4% and sulphour 0.6%. As can be seen, *Amygdalus Scoparia* shells contain a high carbon content which makes them a suitable material from which to obtain activated carbon.

The yield of activated carbon, which is an indication of the activation process efficiency, is the amount of activated carbon produced at the end of the activation step. Total ash content of the resultant activated carbon was determined by ASTM D2866-94 method. The surface area of the PASAC and ZASAC were measured by BET (Brunauer-Emmett-Teller nitrogen adsorption technique). The Iodine number, defined as the mg of iodine per gram of carbon, was determined by ASTM D 4607–94 method. The residual lead concentration was then measured using a flame atomic absorption spectrometer. The pH point of zero charge (pHpzc) indicates the acidic or basic character of the carbon surface [19]. According to the following procedure described by Noh and Schwarz, known amounts of carbon were added sequentially to a given volume of aqueous 0.1 eq $L^{-1}$ NaCl until the pH of solute ceased to change with the further addition of carbon. This value (pHpzc) corresponds to the concentration of $H^+$ ions after all acid groups present on the surface reach to

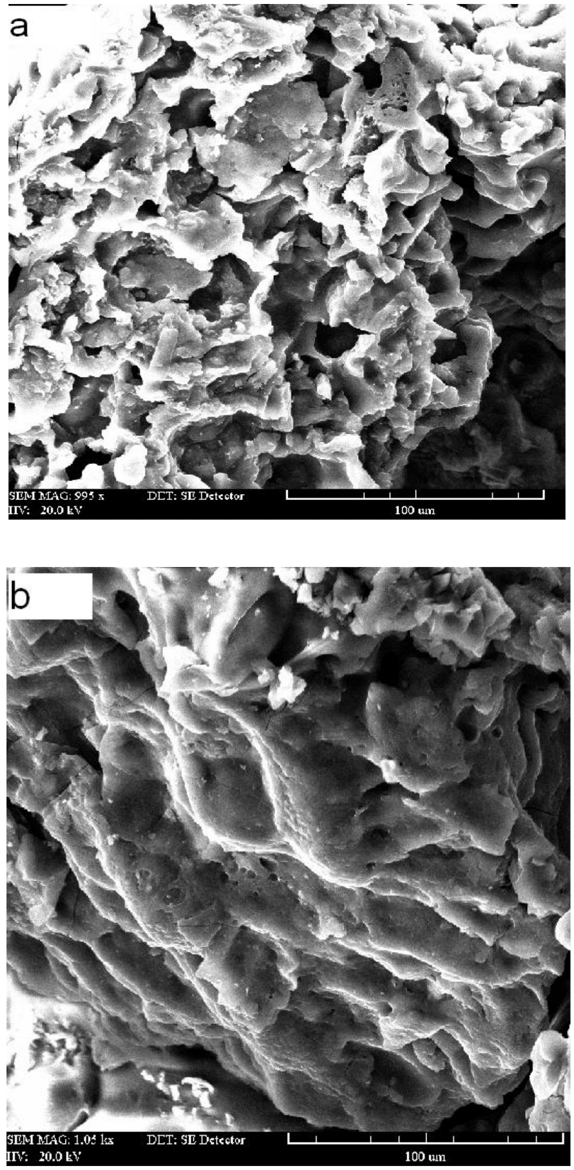


**Figure 2.** SEM images of (a)PASAC and (b) ZASAC (magnification: 1000x).

their dissociate-associative equilibrium. Characteristics of activated carbon are presented in Table 1.

The microstructure of the PASAC and ZASAC were observed by Scanning Electron Microscopy (SEM) (Cam Scan MV2300) and is shown in Fig. 2. Scanning electron micrographs were recorded without sample coating with 1000x magnification. This figure shows that the adsorbent had an irregular and porous surface, indicating relatively high surface areas. This observation is supported by the BET surface area of the activated carbon.

Chemical characterization was studied by Fourier transform infrared (FTIR) spectroscopy in order to

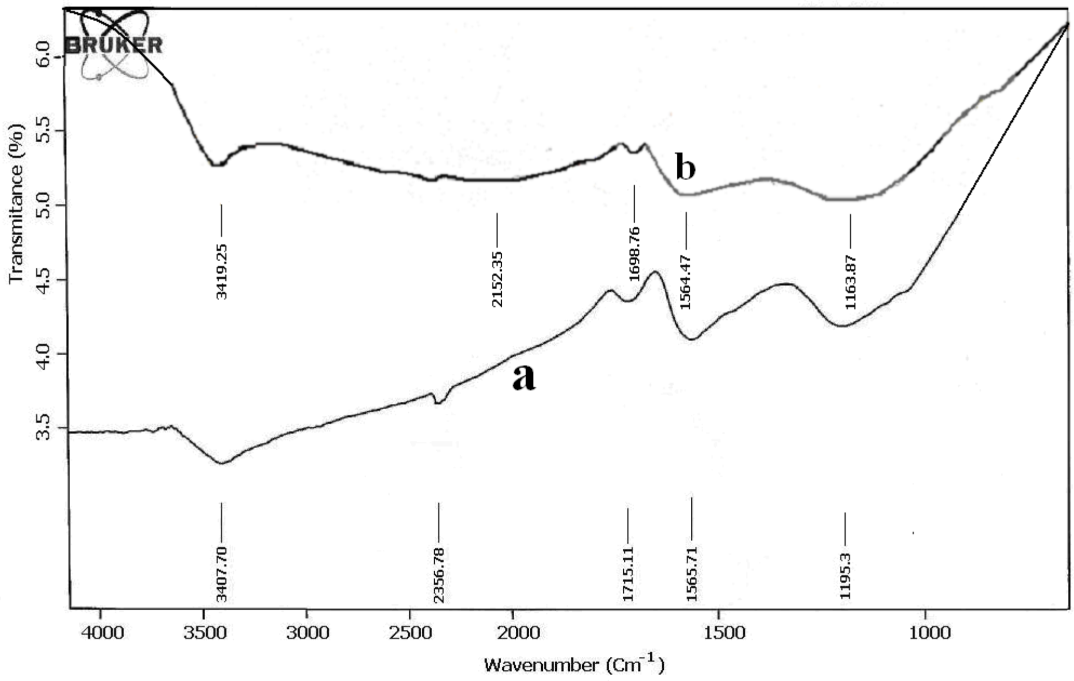


**Figure 3.** Fourier transforms infrared spectra of (a) PASAC and (b) ZASAC.

identify the functional groups at the surface of the PASAC and ZASAC. FTIR spectra were recorded (Fig. 3) with a Brucker Tensor 27 spectrometer (Deutschland, Germany), from 400 to 4000 $cm^{-1}$, using the KBr wafer technique. Wafers were prepared from the mixture of 1 mg of the sample and 100 mg of KBr.

## 2.4. Adsorption experiments

Batch experiments of adsorption were performed in 250 mL Erlenmeyer flasks, with the flasks being agitated on an IKA stirrer model KS (Laboratory Equipment, Germany) at 240 r $min^{-1}$ for identified time intervals. The effect of contact time, solution pH, and initial concentration of lead were studied. Each experiment was carried out by suspending 0.2 g of sorbent in 50 mL of sorbate solution in an Erlenmeyer flask under the optimum conditions determined for the experiment. As pH is a critical parameter in the process, the pH of each solution was adjusted by adding HNO3 or NaOH to pH = 6.0±0.1 before each run. Finally, the supernatant liquids were filtered and the metal concentration in each flask was determined using a flame atomic absorption spectrophotometer.

The effect of pH and contact time on the adsorption of Pb(II) by the PASAC and ZASAC at different pHs and time points were studied using 50 mL of Pb(II) 25 mg $L^{-1}$ at room temperature. The optimum pH and time for the adsorption process was confirmed from the above experiment.

The uptake of metal ions in solution was calculated by the differences in their initial and final concentrations. Each experiment was repeated twice and averaged values are given as the results. The obtained data were employed to calculate the equilibrium metal uptake capacity according to Eq. 1,

**Table 1.** Characteristic of the PASAC and ZASAC.

| Parameters | Adsorbent | |
|---|---|---|
| | PASAC | ZASAC |
| BET surface area ($m^2$ $g^{-1}$) | 959 | 838 |
| Iodine number (mg $g^{-1}$) | 695 | 718 |
| $pH_{pzc}$ | 2.75 | 3.4 |
| Yield (%) | 47 | 40 |
| Total ash (%) | 1.3 | 1.5 |

**Table 2.** Effect of the initial concentration for the adsorption of lead ions onto the PASAC and ZASAC

| Initial Concentration (mg $L^{-1}$) | Adsorbents | | | | | |
|---|---|---|---|---|---|---|
| | PASAC | | | ZASAC | | |
| | $C_e$ | $q_e$ | Removal % | $C_e$ | $q_e$ | Removal % |
| 50 | 0.804 | 3.936 | 98.4 | 2 | 3.84 | 96 |
| 100 | 1.621 | 7.870 | 98.3 | 5.55 | 7.556 | 94.4 |
| 150 | 2.594 | 11.792 | 98.2 | 14.449 | 10.844 | 90.4 |
| 200 | 4.932 | 15.605 | 97.5 | 22.046 | 14.234 | 89.0 |
| 300 | 10.502 | 23.160 | 96.5 | 33.99 | 21.28 | 88.7 |

**Table 3.** Freundlich and Langmuir isotherm constants for the lead adsorption on the PASAC and ZASAC adsorbent

| Adsorbent | Langmuir | | | Freundlich | | |
|---|---|---|---|---|---|---|
| | $Q^0$ | b | $R^2$ | $K_f$ | 1/n | $R^2$ |
| PASAC | 36.63 | 0.162 | 0.9831 | 5.276 | 0.673 | 0.965 |
| ZASAC | 28.74 | 0.057 | 0.801 | 2.633 | 0.56 | 0.983 |

**Table 4.** Comparison of adsorption capacity with other adsorbents

| Adsorbent | $Q^0$ (mg g$^{-1}$) | Ref. |
|---|---|---|
| Siderite | 12.43 | 22 |
| Date pits carbon | 30.66 | 23 |
| Kaolinite clay | 19.27 | 24 |
| Rice husk ash | 12.63 | 25 |
| PASAC | 36.63 | Present work |
| ZASAC | 28.74 | |

$$q_e = v(C_0-C_e)/m \qquad (1)$$

where $q_e$ (mg g$^{-1}$) is the equilibrium amount of metal in the adsorbed phase, $C_o$ and $C_e$ are the initial and equilibrium concentrations of metal ion (mg L$^{-1}$) in the aqueous solution, v is volume of the solution (L), and m is the sorbent dose (g) in the mixture. Isotherm studies were performed using different concentrations of Pb(II) (50-300 mg L$^{-1}$) at room temperature.

The percentage of removal of Pb(II) ions (Re%) in solution was calculated using Eq. 2

$$Re\% = [(C_0-C_e)/C_0] \times 100 \qquad (2)$$

# 3. Results and Discussion

## 3.1. Effect of pH on Pb(II) adsorption

The pH of the aqueous solution is an important factor, as it influences the metal speciation in aqueous solutions as well as the surface properties of the adsorbent, and therefore can affect the extent of adsorption [20]. Thus the adsorption behavior of Pb(II) onto the PASAC and ZASAC was investigated over a pH range of 2.0-8.0 at room temperature (Fig. 4). Higher pH values were not tested because lead hydroxide could be formed as a solid phase precipitate.

The percentage of Pb(II) adsorption on the PASAC and ZASAC increased with increasing pH and reached a plateau value at the pH range of 4.0–6.0 and 5.0–6.0, respectively. Below and above these pH values, the adsorption was decreased.

The pHzpc plays an important role in the adsorption process. At pH values higher than pHzpc, the surface of activated carbon is negative and there is a strong electrostatic attraction between surface groups and Pb(II) species. As a result, the adsorption of Pb(II) was found to be high above pHzpc. The decrease in the adsorption process observed at pH values less than pHzpc, could be attributed to the increase in competition

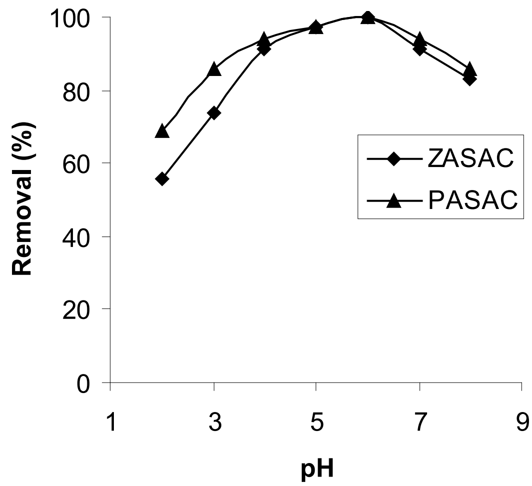


**Figure 4.** The effect of pH on the adsorption of Pb(II) ions onto (a) PASAC and (b) ZASAC. Conditions: 50 mL Pb(II) 25 mg L$^{-1}$, agitation time: 1 h, agitation speed: 240 r min$^{-1}$, sorbent: 0.1 g.

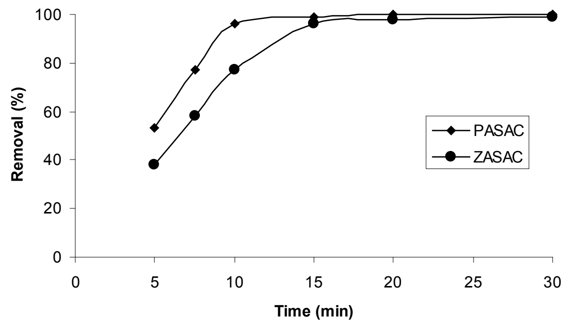


**Figure 5.** The effect of contact time on adsorption Pb(II) onto (a) PASAC and (b) ZASAC. Conditions were the same as Fig. 4, except to agitation time.

between protons and Pb(II) species for the adsorption sites.

## 3.2. Effect of contact time

The effect of contact time on the adsorbed amount of Pb(II) by activated carbonwas studied for times ranging from 5 to 30 min. Fig. 5 shows the effect of contact time on the adsorbed amount of Pb(II) by PASAC and ZASAC from a solution with an initial Pb(II)concentration of 25 mg L$^{-1}$ at room temperature. The adsorption for both PASAC and ZASAC increased sharply with contact time. However, the rate of adsorption of Pb(II) on PASAC was more rapid compared to ZASAC, as it reached equilibrium after 5 min by PASAC, whereas it took 10 min in the presence of ZASAC to reach equilibrium.

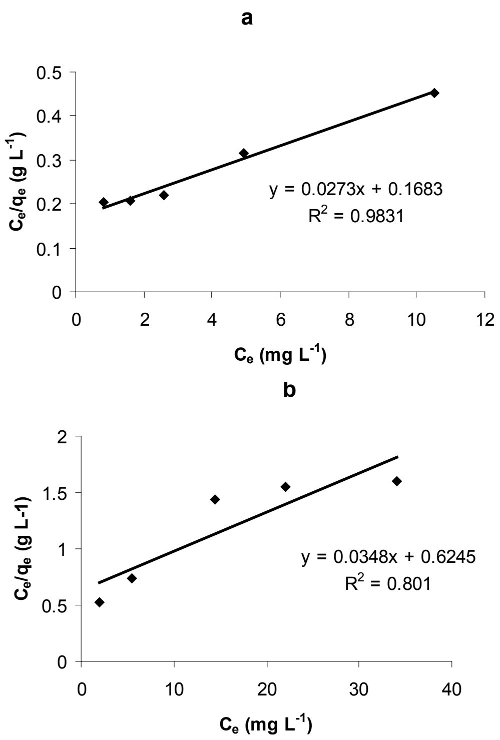


**Figure 6.** Langmuir adsorption isotherms of Pb(II) ions onto (a) PASAC and (b) ZASAC.

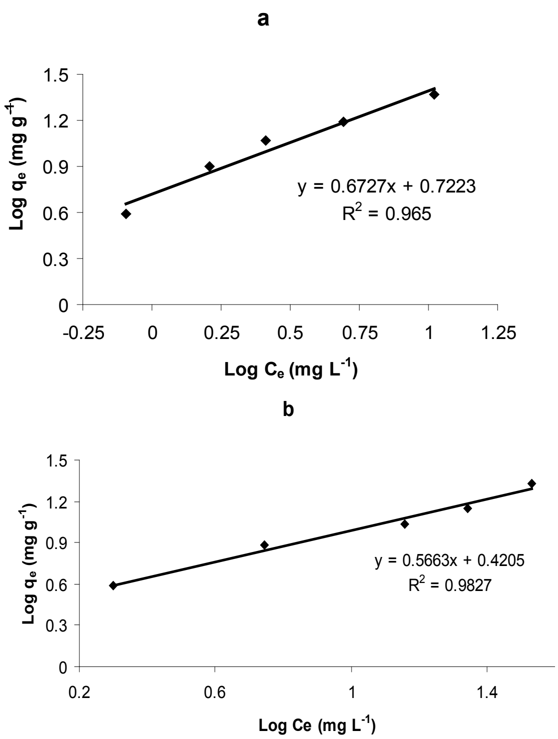


**Figure 7.** Frunlich adsorption isotherms of Pb(II) ions onto (a) PASAC and (b) ZASAC at room temperature.

## 3.3. Initial concentration

Table 2 illustrates the dependency of the process of lead adsorption by the adsorbents on different initial concentrations (50-300 mg $L^{-1}$). For instance, at the lowest concentration (50 mg $L^{-1}$), the amounts of adsorbed lead were 98.4% and 96.0% by PASAC and ZASAC, respectively. The examination of the data also reveals that the amount of adsorbed lead increases with the concentration of the solution, but the percentage of adsorption decreases. These data suggest that the removal of lead ions is highly concentration-dependent. At lower concentrations of lead ions, the number of lead ions which are available in the solution is less compared to the available sites on the adsorbent. However, at higher concentrations the available sites for adsorption become fewer and the percentage removal of lead ions depends on the initial concentration.

## 3.4. Surface chemistry

FTIR studies confirmed the presence of oxygenated functional groups in the PASAC and ZASAC. The FTIR spectrum of PASAC and ZASAC are shown in Fig. 3. The bands at about 1560 $cm^{-1}$ and 1718 $cm^{-1}$ are attributed to ν(C=O) vibrations in the carboxyl groups. The bands at about 3300 and 1000-1220 $cm^{-1}$ are attributed to ν(O–H) vibrations in the hydroxyl and phenolic groups. The location of the bands for hydrogen-bonded OH groups are usually in the range of 3200–3650 $cm^{-1}$ for alcohols and phenols. The appearance of bands between 900 and 1300 $cm^{-1}$ could be assigned to C–O stretching vibrations. Three bands at about 1200, 1700 and 3300 $cm^{-1}$ are attributed to the carboxylate group. From the above analyses, the main oxygen groups present in the PASAC and ZASAC are the carbonyl groups, ethers, alcohol, carboxyl and the phenolic groups.

## 3.5. Adsorption isotherms

Several models have been published in the literature to describe experimental data of adsorption isotherms. The Langmuir and Freundlich models are the most frequently used models. In this work, both models were used to describe the relationship between the amount of Pb(II) ions adsorbed and its equilibrium concentration in solution at room temperature for 1 h (see Figs. 6 and 7).

### *3.5.1. Langmuir isotherm*

The main assumption of the Langmuir method is that adsorption occurs uniformly on the active part of the surface, and when a molecule is adsorbed on a site, the latter does not have any effect upon other incident molecules.

The Langmuir equation can be written as:

$$q_e=Q^0bC_e/(1+bC_e) \quad \text{(Non-linear form) (3)}$$

$$C_e/q_e=(1/Q^0b)+(1/Q^0)C_e \quad \text{(Linear form) (4)}$$

Where $q_e$ is the amount of solute adsorbed per unit weight of adsorbent (mg $g^{-1}$), $C_e$ the equilibrium concentration of solute in the bulk solution (mg $L^{-1}$), $Q^0$ is the monolayer adsorption capacity (mg $g^{-1}$) and b is the constant related to the free energy of adsorption ($b \propto \exp(-\Delta G/RT)$). The constants of the Langmuir isotherm are obtained by plotting $C_e/q_e$ *versus* $C_e$.

The essential characteristics of the Langmuir isotherm can also be expressed in terms of a dimensionless constant of separation factor or equilibrium parameter, $R_L$, which is defined as [21]:

$$R_L=1/(1+bC_0) \quad (5)$$

where b is the Langmuir constant and $C_0$ is the initial concentration of Pb(II) ion. The $R_L$ value indicates the shape of isotherm. An $R_L$ value between 0 and 1 indicates favorable adsorption, while $R_L \geq 1$, and $R_L=0$ indicates unfavorable, linear and irreversible adsorption isotherms. The $R_L$ at different concentrations between 0 and 1 indicates a highly favorable adsorption.

#### 3.5.2. Freundlich isotherm

The Freundlich isotherm can be written as:

$$q_e=K_fC_e^{1/n} \quad \text{(Non-linear form) (6)}$$

$$\log q_e=\log K_f+(1/n)\log C_e \quad \text{(Linear form) (7)}$$

Where $K_f$ is the constant indicative of the relative adsorption capacity of the adsorbent (mg $g^{-1}$) and 1/n is the constant indicative of the intensity of the adsorption. The constants of the Freundlich isotherm are obtained by plotting log $q_e$ *versus* log $C_e$.

Freundlich and Langmuir constants have also been obtained and are given in Table 3. The sorption isotherms were determined at room temperature for a concentration range of 50-300 mg $L^{-1}$. In all different solutions a fixed dose of adsorbent (0.2 g) was used.

### 3.6. Lead desorption

The desorption of Pb(II) ions from the PASAC and ZASAC were carried out by shaking them with different reagents such as HCl, $HNO_3$, and $H_2SO_4$. More than 90% of lead was desorbed with 0.25 mol $L^{-1}$ HCl.

## 4. Conclusion

The present study demonstrates that the activated carbon prepared from *Amygdalus Scoparia* shell, an agricultural waste, is an effective adsorbent for the removal of Pb(II) from aqueous solutions. The adsorption was found to be greatly dependent on the pH of solution as well as initial concentration. The equilibrium data were described satisfactorily by the Langmuir and Freundlich isotherms models and the maximum adsorption capacity of Pb(II) on the resultant activated carbons were determined to be 36.63 and 28.74 mg $g^{-1}$ with $H_3PO_4$ and $ZnCl_2$, respectively. The desorption studies show that the spent PASAC and ZASAC can be effectively regenerated easily for further use. The low cost and rapid adsorptive and regenerative abilities of this sorbent make it a promising technique for cleanup of industrial waste waters. The adsorption capacities of other adsorbents [22-25] were compared with those utilized in the present study and are presented in Table 4. The adsorption capacities of PASAC were greater than the reported values of other adsorbents.

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