Agro-based carbon for lead removal from solutions

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Abstract. The current study utilizes the carbon derived from the lemon peel (CLP) as an adsorbent to remove Pb (II) ions from solutions. Activation of the lemon peels was carried out by adding 1 part of the lemon peels to 1.8 parts of concentrated sulphuric acid, and well-mixed before heating the mixture at a temperature of 150 °C for 24 hours. The produced material, CLP, was employed for lead adsorption from water. A number of parameters namely, treatment times, pH, and doses of CLP were studied to assess their influence on the removability of Pb (II) by the CLP. The results indicated that the optimum contact time, pH, and dosage values for the best removal of lead were 80 min, 5, and 3 g/L respectively. To analyze the observed data gained from batch equilibrium tests, isotherm models (Freundlich and Langmuir), kinetic models (the pseudo-first-order, and the pseudo-second-order), and inter-particle diffusion using non-linear regression techniques were applied for this purpose. The obtained results proved that the equilibrium data have reasonable and good fitness and correspondence with the Freundlich isotherm models. The highest adsorption capacity and the highest removal percentage for Pb (II) were 32.98 mg/g and 99% respectively at ambient temperature.

1. Introduction
The Planet of Earth is facing a water shortage and pollution phenomenon mainly due to the human activities that result in the production of large volumes of wastewaters [1, 2], where the literature shows a significant increase in the concentration of organic matter [3-5], dyes [6-8], biological pollutants [9-11], nutrients [12-14], and heavy metals [15, 16] in freshwater bodies. Besides, the continuous increase in the global temperature has increased the consumption of water and the natural distribution of rain around the world [17-19], which contributed to the pollution of water [20-22] and to change the distribution of the global
population [23-27]. A respected number of forecasting studies demonstrating that more than 30% of the global population will face a lack of drinking water because of the mentioned reasons [28-30]. Heavy metal pollution of water is a serious form of pollution as it affects human health even at low concentrations and it lasts for a long time in water bodies [31, 32]. For example, lead exists commonly in wastewater streams of many different industries as batteries manufacturing, pigments industry, printing, dyestuff, and leaded glass. The high lead concentrations have hazardous impacts on human health, such as liver and kidney damage, hemoglobin reduction, mental retardation, fertility problems, and pregnant women abnormalities. Due to its properties, as it is not degrading biologically and accumulates in the living organism, it has to be removed from water and wastewater. Many different approaches of heavy metal removability from wastewater/water streams have been used including chemical treatment, ion-exchange, electrocoagulation, membranes technique, and adsorption [33-40]. Some of these studies are very efficient, like the electrocoagulation method that is labeled by the low energy consumption and low production of sludge [37, 41], which means less use of lands as landfills for the dumping of sludge [42-46] and consequently better cost-effectiveness. Additionally, the produced sludge from this method could be reused in many fields, such as mortar [47, 48], concrete [49, 50] and other construction materials [51-53]. However, recent studies are looking for recyclable materials to adsorb heavy metals from water. Adsorption technique showed high affectivity for the elimination of heavy metal pollution from solutions by utilising a low-cost adsorbent [54-56]. Waterworks sludge and Waste Foundry Sand have been explored as cost-effective adsorbents due to their potential use and their ability for heavy metal uptake [57, 58]. The composition of the peel of the citruses plant is consists of water and solids at percentages of 80-85% 20-15%, respectively. The solid part is made from pectin, cellulose, sugar, phenols, and protein. The maximum plentiful type of biomolecule in citruses’ peel is the polysaccharide. In addition to that, the occurrence of pectineus matter offers potential transformations of enzymes or chemicals to produce its favourite characteristics, like ion-exchange capacities, galacturonic acids, the major sugars content in citruses pectineus matter. The current work depicts the batch adsorption’s characteristics of Pb (II) on carbon derived from lemon peel focusing on different parameters; such as contact periods, pH-value, and dosages on adsorption capacity. Adsorption isotherms and kinetics have been used to analyse the observed data.

Adsorption models relate the sorbed chemicals on solid materials ($q_e$, mg per g) with the final concentrations of these chemicals ($C_e$, mg per L). It is calculated at controlled levels of temperatures and pH; it usually shows a good fitness with one or more of isotherm models, such as Freundlich or Langmuir ones [59].

### 1.1. Freundlich Model

This model is used for the description of the sorption of multilayer and surfaces of heterogeneous; its general formula could be represented as follows [59]:

$$q_e = K_F C_e^{1/n}$$  \hspace{1cm} (1)

$K_F$: The maximum amount of the adsorbed chemical.  
$1/n$: is the sorption’s strength, and it is always less than 1.0.

### 1.2. Langmuir Model

The following developed formula of the Langmuir model is suitable for the homogenous surface and monolayer sorption [59]:
\[ q_e = \frac{q_{\text{max}} \cdot b \cdot c_e}{1 + b \cdot c_e} \] (2)

\[ q_{\text{max}}: \text{The highest adsorption capacities (mg per g)}. \]
\[ b: \text{The intensity of the contaminant to the solid phase}. \]

1.3. Kinetic Models
To design the suitable sorption process, the rate of the transferred solute from the aqueous phase to the solid phase is utilized for this purpose [59]. This rate is estimated using the following formula of kinetic models:

1.4. Pseudo First Order Model
Formula 3 is used to describe the sorption rates as a function of the time:

\[ \frac{dq}{dt} = k_1 (q_e - q_t) \] (3)

By applying \( q_t = q_e \) at \( t = t \) and \( q_t = 0 \) at \( t = 0 \), Eq.3.3 is integrated to produce the following model:

\[ \ln(q_e - q_t) = \ln q_e - k_1 t \quad \text{or} \quad q_t = q_e (1 - e^{-k_1 t}) \] (4)

where \( q_t \) (mg per g) is the amounts of contaminants sorbed on the solid matrix at time \( t \) and \( q_e \) (mg/g) is equilibrium times, and \( k_1 \) is Pseudo first order constant (1/min).

1.5. Pseudo Second-Order Model
This model was developed basing on a number of assumptions that could be summarised as follows: the monolayer of contaminants accumulated on the surface of sorbent, and the sorption energies remained the same for sorbents and sorbed chemicals. This model is shown in the following equation:

\[ \frac{dq}{dt} = k_2 (q_e - q_t)^2 \] (5)

\( k_2: \text{The Pseudo second order constant (g/mg.min)}. \)

Integration of Eq.5, at identical conditions to that in the previous models, led to the following form:

\[ \frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t \quad \text{or} \quad q_t = q_e \left(\frac{t}{\frac{1}{k_2 q_e} - \frac{1}{q_e}}\right) \] (6)

2. Methodology

2.1. Adsorbent and contaminants
Lemon peel was washed twice using tap water and deionized water respectively to get rid of dust and other contaminants. After that, it was dried by sun rays and used to develop activated carbon. Activation was completed by mixing 1 weight unit of the lemon peel with 1.8 weight unit of concentrated sulphuric acid. An oven was used to dry the mixed materials at 150 °C for 24 hours. For free acid removal, deionized water was used to wash the carbonized material several times and then this material was dried again at 105 °C. This material was employed for lead adsorption. The adsorbent’s particle size was ranging from 0.5–0.8
mm. A surface analyser (Poresizer-micro metrics 9320) was utilised to estimate the surface area and porosity. The adsorbent’s physical properties including Bulk density (g/mL), Ash content (%), Moisture content (%), Surface area (m²/g), and Porosity (ml g⁻¹) are 1.02, 5.8, 6.2, 367, and 0.59, respectively. The stock solutions of polluted water, 1000 mg/L of lead, was synthesized by dissolving 1.599 g of lead(II) nitrates in distilled water in a flask with a volume of 1L. To adjust the pH to the desired value, 0.1 M nitric acid or 0.1 M Sodium-hydroxide was be added to this solution. The solution is used subsequently to synthesize 80 mg/L of the lead solution by dilution with water.

2.2. Batch Study

Batch tests were adopted to obtain the required data on equilibrium and kinetics of interactions between lead (II) and the prepared sorbents. The operational parameters, including time, initial pH, and sorbent dose, were studied to attain the highest removal efficiency of lead. The needed number of batch tests were conducted using 100 mL of a solution that has an initial concentration of lead of 80 mg/L, which was placed in a 250 ml flask. The addition of different dosages of sorbent to flasks was conducted during the agitation process, at 150 rpm, for 80 min. Then, the filtration processes were carried out for the solution of each flask to isolate the solids. The remaining concentrations (C_e) of the Pb(II) in filtered solution were measured using atomic absorption spectroscopy (Sens AA, Japan). The principle of mass balance was used to calculate the amount of sorbed pollutants per unit mass of the sorbent. Sorption studies were carried out with contact time ranging from 20 min to120 min and pH values from 2 to 7. For optimum conditions, the amounts of the sorbed pollutants on the sorbent (q_e) was calculated using (Wang et al., 2009):

\[
q_e = (C_o - C_e) \frac{V}{m} 
\]  

(7)

where V: volume of water (L), and m: the sorbent weight (g). The adsorption isotherm has schemed between calculated q_e and C_e. The removal efficiency (R) was determined according to the differences between C_o and C_e as follows:

\[
R = \frac{(C_o-C_e)}{C_o} \times 100 
\]  

(8)

3. Results and discussion

3.1. Dosage of CLP
The dependence of lead adsorption on the adsorbent doses was investigated by adding different amounts of CLP (from 1 to 4 g/L) at 25°C, while the \( C_0 \), pH, agitation speed, treatment time were kept constant at 80 mg/L, 5, 150 rpm, and 1 h, respectively. The Pb(II) removability, as a function of different doses of CLP, is presented in Figure 1. It can be noticed that the removability of the CLP enhanced when the adsorbent dose increased from 1 to 3 g/L at a constant initial concentration of Pb. This can be explained by increasing the available active sites due to the higher amounts of adsorbent in the solution. In addition, the maximum adsorption process starts after adding a certain dose of adsorbent (1 g) hence the amount of Pb(II) engaged to the adsorbent. With the addition of extra amounts of adsorbent, the Pb(II) concentration in solution remains constant. Due to the low difference between the adsorption at doses of 3 and 4 g, the rest of the experiments were conducted using 3 g of CLP.

3.2. Equilibrium Time

One of the batch test requirements is to keep the contact time constant to attain the equilibrium concentration. Hence, the influences of contact time on the removability of Pb(II), using 3 g/L of CLP, was studied using batch tests at 25°C, and the results are shown in Figure 2. This figure clarifies that Pb(II) removal efficiency dramatically increased with the contact time. It can be noticed that at the initial stage the sorption rate was fast but after that, it began to slow down gradually. This slowness is explained by the diminution in the numbers of sorption sites on the adsorbent surface. At a contact time of 80 min, about 100% Pb(II) removal was achieved however, with further contact time increase (more than 80 min) the Pb(II) concentrations relatively remained constant. This means that the residual Pb(II) concentrations did not change remarkably after a contact time of 80 min. Therefore, all sorption experiments were conducted using this contact time.
3.3. Initial pH of the solution

The sorption capacity for lead ions from solution is affected by the pH level of solutions because of its influences on the characteristics of the sorbent surfaces and the ionic formula of the contaminants in the solution. Additionally, the sorption behaviour of any sorbents for metal ions is affected by the pH value due to its effects on the protonation and deprotonation, sorbent surface, hydroxides formation of metal, and the interaction between metal ions and sorbents [59]. Therefore, the sorption of Pb(II) on CLP was tested as a function of the initial pH values (from 2 to 7) for a constant CLP dosage of 3 g/L at a constant initial lead concentration of 80 mg/L and agitation speed of 150 rpm (Figure 3). This figure shows that the sorption behaviour of metal ions is changed with the change of pH values. It can be noticed that the Pb(II) sorption of lead reached the best level at pH of 5, which can be explained by decreasing the competitions between protons and metals for the surface sites, and the decreasing of the positive surface charges, which leads to a lower repulsion of the sorbing metal. At pH values, less than 5, only the occurrence of the dissociated aqua-ion-forming Pb(II) ions is found and no hydroxy complexes in the solution. at low pH value, the competition between the proton and metal ions to occupy the active binding sites leads to a decline in the metal sorption process as all the binding sites may be protonated at low pH value (around 2). In addition, the progressive decline of Pb(II) removability was observed when pH values increased above 5, and this decline can be explained by the competition of formed hydroxyl groups with Pb(II) ions for CLP occupation. At high pH values, the charge of the CLP surface turns to be negative, hence the repulsion forces between Pb(II) and this surface become stronger. That is why a pH of 5 was adopted in this study.

Figure 2. Effects of times of the removals of Pb(II) by CLP.
Kinetic and Isotherm analysis

A good fitness of kinetic data found with the Pseudo-first order, Pseudo-second order, and Weber – Morris models as depicted in Figure 4. To calculate the constants of these models, non-linear regressions methods were used, as itemised in Table 1. Obviously, the sorption of Pb(II) fits the Pseudo-second order kinetic model, which indicates the predominance of the chemical sorption mechanism on the sorption process [59]. These tasks were commenced using the intra-particles diffusions as the sorption mechanism identification which based on the kinetic models presented before cannot be attended. This model depends on the theory proposed by Weber and Morris (1962) [59]. This model is an empirical formula where sorbed quantity is variable as a function of $t^{1/2}$ rather than $t$ as shown below:

$$q_t = k_{int} t^{0.5} + C$$  \hspace{1cm} (9)$$

where $k_{int}$ (mg/g hr$^{1/2}$) represents the rate constant of stage $i$ and it is equal to the slope of the relationship that relates $q_t$ with $t^{1/2}$. Similarly, $C$ equals the intercept of stage $t$, and it indicates the thicknesses of boundary layers. This means that the boundary layer becomes more effective as the value of the intercept grows larger. The intra-particle diffusion takes place when the correlated relationship between $q_t$ and $t^{1/2}$ becomes linear. If the linear plot goes through the origins, the rate-limiting processes is equal to intra-particles diffusions, otherwise, other mechanisms must be considered. In general, the adsorption mechanism is considered to include: (i) mass transformation of adsorbates from the bulk phases to particles’ surfaces, (ii) adsorptions processes on the surface sites, and (iii) intra-particles diffusions of the molecule of adsorbates to adsorption sites by pored diffusions and/or surfaces diffusions mechanisms. Step (ii) is usually assumed to be very fast, as a result, that the big molecule, with elongated treatment time to the equilibrium, is always adsorbed by diffusion as external films’ resistances and/or internal diffusions mass transports or intra-particles diffusions control it. To find if intraparticle diffusion controls an adsorption process, a classical approach is used for that by plotting the adsorbed amount versus the square root of time, $t^{1/2}$. If the plot is linear and goes through the origin point, that means the intraparticle diffusion is the controller of the adsorption process. Figure 4 illustrates the adsorbed amount of Pb(II) as a function of $t^{1/2}$ for the adsorbents. It can be noticed that the intraparticle diffusion model has a satisfactory fitness with the observed data, attending a linear section that doesn’t go through the origin refers to the absence of intraparticle diffusion control on the tetracycline adsorption on these adsorbents.

Sorption tests are implemented with a pH of 5, speed of 150 rpm, and contact time of 80 min for different concentrations. The Freundlich and Langmuir isotherms are shown graphically in Figure 5 and Table 1 lists
their constants that are calculated by using nonlinear fitting of these models with sorption measurements through the application of the ‘Solver’ option in the Microsoft Excel 2016. Based on this figure and coefficient of determination ($R^2$) in Table 1, Freundlich isotherm is better than the Langmuir model in the description of sorption measurements.

| Model                     | Parameter   | Value          |
|---------------------------|-------------|----------------|
| **Freundlich**            | $K_F$ (mg/mg)(L/mg)$^{1/n}$ | 27.41692926    |
|                           | $n$         | 18.52061845    |
|                           | $R^2$       | 0.991885257    |
|                           | SSE         | 0.173662333    |
| **Langmuir**              | $q_{max}$ (mg/g) | 32.97901516    |
|                           | $b$ (L/mg)  | 8.127462466    |
|                           | $R^2$       | 0.924008622    |
|                           | SSE         | 2.523859786    |
| **Pseudo-first order**    | $q_e$ (mg/g) | 40.89433729    |
|                           | $k_1$ (1/min) | 0.040782145    |
|                           | $R^2$       | 0.98408137     |
|                           | SSE         | 3.756432876    |
| **Pseudo-second order**   | $q_e$ (mg/g) | 48.78247183    |
|                           | $k_2$ (g/mg min) | 0.000993473    |
|                           | $R^2$       | 0.959178276    |
|                           | SSE         | 9.729970533    |
| **Intra-particle diffusion** | $k_{lin}$ (mg/g min$^{0.5}$) | 2.6196    |
|                           | $R^2$       | 0.8417         |
4. Conclusion
The possibility to use carbon derived from the lemon peel (CLP) for Pb(II) removability was investigated. It was found that carbon derived from the lemon peel (CLP) showed high affectivity for Pb(II) removal from aqueous solution, the maximum adsorption capacity for Pb(II) was 32.97901516mg/g. The percentage of removal efficiency was dramatically affected by the treatment times, initial pH of the solutions, and adsorbent dosages. The process kinetics followed the pseudo-second-order model. Thus, it could be concluded from the results of the commenced experiments that recycling of bio-wastes in water treatment could be an eco-friendly and economical effective alternative for the traditional expensive adsorbents.
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