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Pb (II) REMOVAL FROM AQUEOUS SOLUTION BY ADSORPTION ON ACTIVATED CARBON FROM KIWI PEEL

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Abstract

A new low-cost activated carbon was developed from kiwi peel for removing Pb(II) from aqueous solution. The adsorbent characteristics were determined by different analyses such as BET, FTIR and SEM. The adsorbent demonstrated remarkable characteristics such as high surface area (306.18 m2.g-1) and large total pore volume (0.4810 cm3.g-1). The effect of pH, contact time, initial concentration, shaking rate and adsorbent dosage on the Pb removal efficiency was investigated. It was found that the optimum pH for removing Pb(II) was 6. The equilibrium experimental data were analyzed by several model isotherms. The adsorption models were found to fit the experimental data in the order of Freundlich> Langmuir >Tempkin. The maximum adsorption capacity of Pb (II) adsorbed by kiwi peel-based activated carbon, was 158.82 µg.g-1. The kinetics of Pb (II) adsorption was investigated using different kinetics models. Experimental results were well fitted with pseudo-second-order model. It could be inferred from the results that kiwi peel is a promising and cost effective raw material for the removal of contaminants from wastewaters.

Key words: adsorbent, isotherm, kinetics, kiwi peel, Pb (II)

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1. Introduction

Heavy metals are known as dangerous contaminants that hurt human health. These contaminants are found in liquid wastes of a number of industries such as plating, paint and dyes, glass operations, Pb batteries, electroplating and mining (Aroua et al., 2008). Since these kinds of contaminants possess strong complexing capacity (which can be assigned to high concentration of organic and inorganic ligands), heavy metals have high tendency to be extracted from dump wastes and diffuse to the groundwater (Petrangeli Papini et al., 2001). Among the heavy metals, Pb is one of the most toxic contaminants, even at low concentrations. It damages the central nervous system, kidneys, liver and it may directly or indirectly cause diseases like anemia and hepatitis (Martins et al., 2006).

Various techniques are available for mitigating the amount of heavy metals in wastewaters such as chemical precipitation, adsorption, ion exchange and membrane separation. Among these techniques, adsorption is considered attractive due to its simplicity and effectiveness in removing heavy metals from dilute solutions. Activated carbon is a highly porous, carbonaceous material which can be applied as special adsorbent (Chen et al., 2002). High adsorption capacity is one of its salient feature which can be used in mixture separation and liquid purification (Covaliu et al., 2016; Jia and Lua, 2008). Any carbonaceous material can be used as an activated carbon through physical and chemical activation.

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Nature of the precursor, activation method and activation conditions have drastic impact on pore size distribution, pore shape and surface chemistry of activated carbon. Physical activation is comprised of two stages: (i) the material is carbonized under inert atmosphere; (ii) the carbonized material is activated at high temperature for developing pore structure using steam or carbon dioxide as an activating agent (Pirzadeh and Ghoreyshi, 2013). The activation reagent is the key factor in chemical activation, where, presence of chemicals leads to pore development. In this method, after impregnation of precursor with activation reagent, the slurry is heated in an inert atmosphere. Oxidation reactions of the chemicals which is followed by dehydration, enhance the pore structure. Then, the residual impurities (char) is detached through repeated washing of produced char (Ioannidou and Zabaniotou, 2007).

As opposed to physical activation, chemical activation is more suitable due to some gains such as low energy consumption, high carbon yields, and considerable surface area (Gratuito et al., 2008; Prahas et al., 2008). Since burn-off char is not required, the total yield of chemical activation tends to be greater (Mouni et al., 2011). Although use of commercially activated carbons are still popular, high cost of production and regeneration restricts the application of these kinds of activated carbons (Li and Wang, 2009; Mane and Vijay Babu, 2012). Thus there is a growing demand to find an adsorbent, not only easily available but also relatively effective and low cost for adsorption of Pb (II) from effluents. The low-cost adsorbent can be prepared from rice husk (Ajmal et al., 1998), pine bark (Al-Asheh and Duvnjak, 1998; Tofan et al., 2012), nutshells (Ahmedna et al., 2004), and olive stone (El-Sheikh et al., 2004), fruit peels (Ponou et al., 2016), leaves (Gunasekar et al., 2017).

In recent decades, rapid urbanization and industrialization have posed extreme pollution to the environment. This matter jeopardizes the environment since these contaminants in wastewaters are non-biodegradable and hardly dissociate via conventional biological treatment. Hence it is difficult to ignore contaminants from wastewaters (Pirzadeh and Ghoreyshi, 2013; Revathi et al., 2012).

According to the global surveys, Iran is one of the greatest kiwi producers in the world. Kiwi peel is a valueless part of kiwi fruit and because of this, it was chosen as a precursor for synthesizing activated carbon. The novelty of the present work lies in using new precursor for synthesizing activated carbon for removing Pb (II) and relevant issues in this area are scarce.

This research addresses the application of a new low-cost adsorbent prepared from kiwi peel. Then, the efficiency of the new adsorbent was experienced for mitigating Pb (II) from aqueous medium based onequilibrium and kinetic studies in a batch system. In addition, the effects of pH solution, adsorbent dosage, contact time, initial concentration and shaking rate were examined.

2. Material and methods

2.1. Preparation of activated carbon

The precursor used for production of adsorbent was kiwi peel which was rinsed with water at first and then exposed to sunlight for two weeks in order to become completely dry. After that, the dried peel crushed to small particles. About 500 g of samples were impregnated with 1000 mL phosphoric acid (89%) and stirred for 5h at 85°C. When the slurry dried completely, sample was placed in a special instrument which could bear high temperature for carbonization. After fixing the cap, the instrument was stabilized in a cylinder and placed in a tube furnace. The cylinder was evacuated because of the reaction that may occur between the oxygen and sample. The carbonization step was carried out by raising the temperature at rate of 12°C/min to 700°C and maintaining the temperature for 1h. The adsorbent which was extruded from the instrument, rinsed with hot water until pH reached to 7. The obtained adsorbent was dried at 105°C for 24 h and then sieved to 0.15-0.85 mm.

2.2. Adsorbent characteristic

The surface morphology was monitored through Scanning Electron Microscopy (SEM, KYKY-EM3200, China). The Fourier Transform Infrared Spectroscopy (FTIR) was carried out on a FTIR460 Spectrophotometer (Jasco-Japan) to determine the nature of functional groups. The N_2 adsorption-desorption isotherm was used to measure textural properties at 77 K (Gemini, USA). The BET surface area was obtained using Brunauer-Emmet-Teller (BET) method. Total pore volume was determined from the volume of liquid nitrogen adsorbed at the relative pressure of 0.995. The pore size distribution was calculated using adsorption-desorption isotherms by the Barrett-Joyner-Halendan *(*BJH) method and micropore volume was obtained according the t-plot method.

2.3. Adsorption experiment

Batch adsorption experiments were carried out in a series of stoppered flasks. A weighed amount (6 g.L^{-1}) of adsorbent was introduced into the flasks (250 mL) containing various concentrations of Pb (II). The total volume of the solution was 50 mL. The pH of the solution was adjusted using dilute 0.1N NaOH or 0.1N HCl. Then, the flasks were placed in an orbital shaker (ORBITAL INCUBATOR– SI5) at room temperature (20°C) to attain the equilibrium. Then, the solutions were filtered and the final Pb (II) concentration were determined using atomic adsorption spectrophotometer (AA240). To evaluate the effect of significant factors on adsorption, parameters such as pH (2-8), contact time (10-180 min), initial Pb (II) concentration (500-1000 μg.L^{-1}) and shaking rate (220, 280 and 340 rpm) were

investigated in batch adsorption system. The removal efficiency of heavy metal can be calculated from (Eq. 1):

$$Removal(\%) = \frac{C_0 - C_e}{C_0} \qquad (1)$$

where C_0 and C_e (mg/L) are initial and equilibrium concentrations, respectively. In all cases the adsorption capacity is calculated from the following (Eq. 2):

$$q_e = \frac{(C_0 - C_e) \times V}{m} \qquad (2)$$

where *V* is the volume of the solution (L) and m is the mass of the adsorbent (g).

3. Results and discussions

3.1 Characterization results

3.1.1. SEM

Surface morphology of activated carbon was sighted by (SEM). Fig. 1 shows the SEM micrograph of the synthesized activated carbon.
As can be seen in Fig. 1, activated carbon made from kiwi peel has well-structured porosity which makes it suitable for adsorption of Pb (II).

3.1.2. FTIR

Fig. 2 (blue line) shows the FTIR spectrum of the kiwi peel derived activated carbon before adsorption. At the broad and flat band appeared at 3420 cm^{-1} could be attributed to the O-H stretching vibrations of hydrogen-bounded hydroxyl group. A peak around 1600 cm^{-1} can be associated to the C=O stretching vibrations of ketones, lactones or carboxyl groups. A peak observed around 1200 cm^{-1} with sharp and strong shoulder distinguished at 1000 cm^{-1} can be ascribed to the phosphoric compounds formed due to
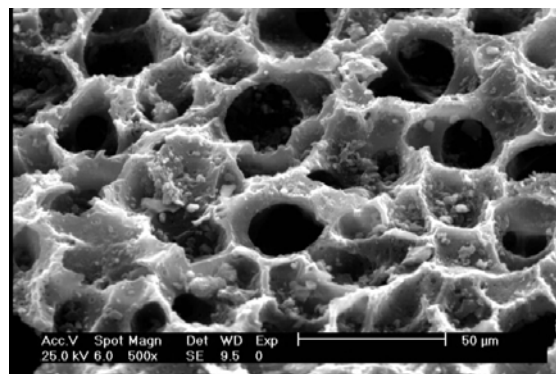
the phosphoric acid activation.

Fig. 1. SEM micrograph of kiwi peel derived activated carbon

Considering this, the peak observed at 1180 cm^{-1} can be associated to the stretching vibrations of hydrogen bounded P=O, stretching vibrations of O-C in P-O-C linkage and P=OOH. The sharp peak at 990 cm^{-1} can be attributed to the ionic bound of P$^+$-O$^-$ in acid phosphate esters and symmetrical vibrations in the chain of P-O-P (Puziy et al., 2002). Fig. 2 (green line) shows the FTIR spectrum of the adsorbent after adsorbing Pb (II). The peaks located at 990, 1180, 1450, 3420 cm^{-1} shifted to 1100, 1210, 1380 and 3400, respectively, which can be ascribed to the Pb (II) adsorption. These shifts can be due to the ion exchange that occurred by carboxylate and hydroxylate anions which propose the great impact and contribution of these acidic groups in adsorbing Pb (II) (Yang and Cui, 2013).

3.1.3. BET surface area

The detail information about textural properties of the kiwi peel-based activated carbon and commercial activated carbon including are listed in Table 1.
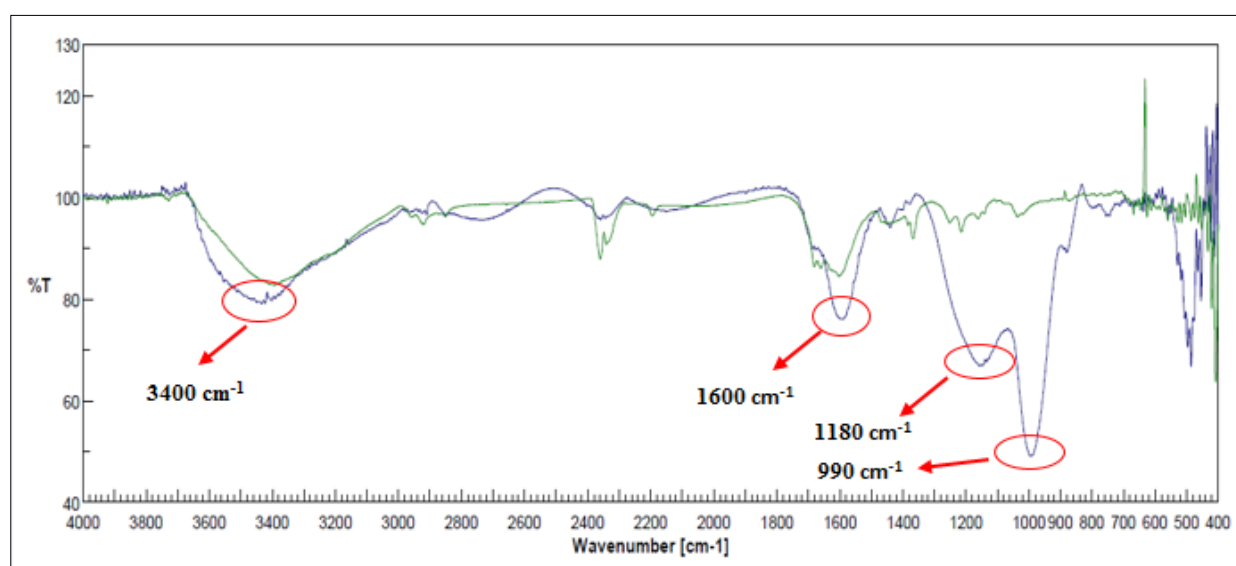

Fig. 2. FTIR spectra of activated carbon; before adsorption of Pb (II) (blue line) and after adsorption of Pb(II) (green line)

Table 1. Textural properties of as-synthesized and commercial activated carbon

Properties	kiwi peel-based activated carbon	commercial coal-based activated carbon
BET surface area ($m^2.g^{-1}$)	306.18	545.27
Total pore volume ($cm^3.g^{-1}$)	0.4810	0.4760
Micropore volume ($cm^3.g^{-1}$)	0.4371	0.2512
Average pore diameter (nm)	5.4131	3.4916

According to IUPAC classification, this activated carbon with average pore diameter of 5.41 nm ($2 \leq d \leq 50$) is classified at mesoporous material.

3.1.4. Effect of pH

Generally, the pH of the solution has a great impact on the uptake of the heavy metals because it affects the surface charge of the adsorbent (Momcilovic et al., 2011). In order to investigate the effect of the solution pH on adsorption of Pb (II) ion, the batch experiments were carried out in the pH range of 2-8. As can be seen in Fig. 3, the adsorption capacity increased as the pH of the solution was increased, especially at pH values greater than 6. This behavior can be attributed to this reason that, at lower pH, the surface of the adsorbent is protonated and there is a competition between the H^+ ions and free Pb (II) and their hydroxides toward the fixation sites (Low et al., 1993). As a consequence, H^+ ions react with the negative adsorption sites of the adsorbent which causes a reduction in available binding sites for adsorption of Pb (II) (Mouni et al., 2011). The great difference in Pb (II) uptake at pH 6 and 7 may be due to the negative charge on the surface of the adsorbent which is increased as the oxygen containing functional groups became more deprotonated with increasing pH (Li and Wang, 2009). It can be also mentioned that as the pH increases to 7, the effect of H^+ decreases. Thus, the interaction between the adsorbent and Pb (II) ions increased with increasing pH in acidic medium. The optimum pH was found around 6 with 50% removal efficiency and adsorption capacity of 39.166 $\mu g.g^{-1}$. It should be mentioned that, according to the Pourbaix diagram of lead, at pH values greater than 6.7, Pb (II) ion precipitates as $Pb(OH)_2$. Therefore, no uptake can be determined above pH=7 (Momcilovic et al., 2011).
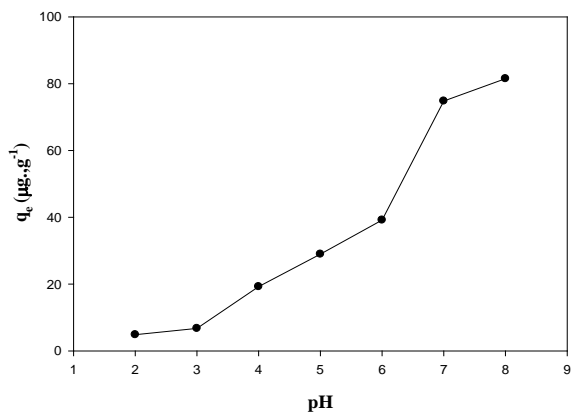

Fig. 3. Effect of pH on Pb (II) removal from aqueous media by activated carbon from kiwi peel,

(at 20°C, C_0=500 $\mu g.L^{-1}$, m=6 $g.L^{-1}$)

3.1.5. Effect of contact time

Effect of contact time on adsorption of Pb (II) onto the synthesized activated carbon at 20°C was studied and the results are shown in Fig. 4. From Fig. 4, the adsorption rate was increasing for the first 70 min because of vacant sites which were available for Pb (II) to interact with. After 90 min, the rate of adsorption became slow due to saturation of active sites. It should be stipulated that the adsorption process usually follows three sequential steps (Faust and Aly, 1983). At first, film diffusion occurs where the adsorbate transfers through the solution to the surface of the particle, then it moves into the inner part by pore diffusion and finally, it is adsorbed into the active sites of the adsorbent particle (Hameed and Rahman, 2008).
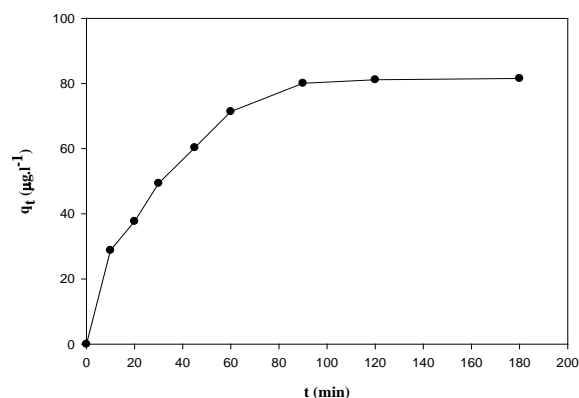

Fig. 4. Effect of contact time on adsorption of Pb^{2+} on kiwi peel derived activated carbon (C_0=500 $\mu g.L^{-1}$, m= 6 $g.L^{-1}$)

Also, to evaluate the performance of the adsorption process for the Pb (II) removal of by the kiwi peel-based activated carbon, same experiment was implemented with a typical commercial activated carbon (code: 1.02183.100) produced from charcoal provided by Merck Co., Germany.

Both experiments were carried out at constant initial concentration of 500 $\mu g.L^{-1}$, adsorbent dosage of 6 $g.L^{-1}$ and temperature of 20°C. Fig. 5 illustrates the removal performance of the commercial activated carbon and the one synthesized in this work. From Fig. 5, it can be understood that although commercial activated carbon showed better performance in the first 100min than kiwi peel-based activated carbon, it adsorbed same amount of Pb (II) as the kiwi peel-based activated carbon did at the end of the process. The sharp rise in the adsorption of Pb (II) in initial times for commercial activated carbon can be attributed to the higher microporosity that commercial

activated carbon had for removing Pb (II) from aqueous media, in the other words, it had more vacant spaces for adsorbate in initial times compared to kiwi peel-based activated carbon.
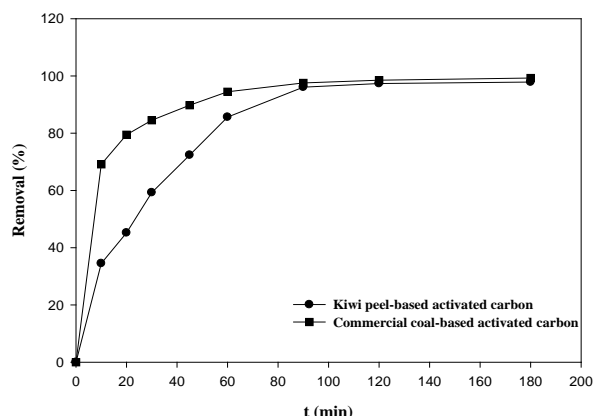
Fig. 5. Pb(II) removal efficiency by commercial activated carbon and kiwi peel-based activated carbon (C_0=500 µg.L^{-1}, m=6 g.L^{-1})

3.1.6. Effect of initial concentration

The effect of initial metal concentration on the adsorption capacity was studied at several initial concentrations of Pb (II) in the range of 500-1000 µg.L^{-1} at optimal pH 6.0 with 6 g.L^{-1} adsorbent at 20°C and contact time of 180 min. Fig. 6 shows that the equilibrium adsorption capacity of Pb (II) increased from 81.551 µg.g^{-1} to 158.821 µg.g^{-1} when the Pb (II) initial concentration increased from 500 to 1000 µg.L^{-1}. An increase in the initial concentration brings stronger driving force which makes adsorbate ions move through the available active sites.
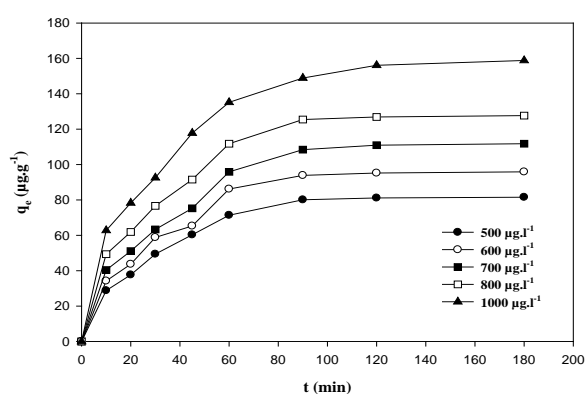
Fig. 6. Effect of initial concentration on Pb (II) adsorption (T=20°C, m= 6 g.L^{-1})

3.2. Shaking rate

Shaking rate can influence the distribution of the solute in the bulk solution (Aroua et al., 2008). Different shaking rates (200, 280 and 340 rpm) were studied and the results showed that increasing the shaking rate did not have a significant effect on adsorption of Pb (II) onto the activated carbon.

Therefore, further experiments were performed at 280 rpm.

3.3. Kinetic models

The adsorption kinetics describes the uptake in terms of time variation (Tan et al., 2009). Pseudo-first order (Febrianto et al., 2009) and pseudo-second order (Ho et al., 2000) kinetic models were used to determine the mechanism of the process. The pseudo-first order kinetic model is expressed by Eq. (3) (Hameed et al., 2007):

$$ln(q_e - q_t) = lnq_e - k_1t \qquad (3)$$

where k_1 (g.µg^{-1}.min^{-1}) is the rate constant of pseudo-first order adsorption, q_t and q_e are amount of metals adsorbed (µg.g^{-1}) at time t (min) and at equilibrium. The values of k_1 and q_e were determined from the slop and intercept of the plot of ln (q_e-q_t) versus t (Fig. 7).

Ho and Mc Kay (Ho and McKay, 1999) have reported that most adsorption systems conforms pseudo-second order kinetic model which is based on equilibrium adsorption (Li et al., 2005). This equation is given by Eq. (4):

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

where k_2 (g.µg^{-1}.min^{-1}) is equilibrium rate constant for pseudo- second order kinetic model; k_2 and q_e were obtained from the slope and intercepts of the plot of t/q_e versus t (Fig. 8).
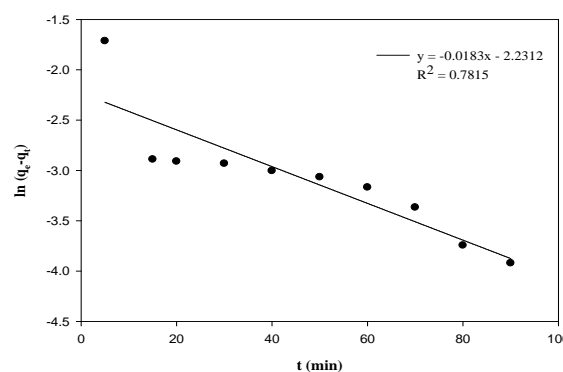
Fig. 7. Pseudo-first order model for adsorption of Pb (II) at 20°C, C_0=500µg.L^{-1}, pH=6

By comparing the correlation values of these models (R^2), it is obvious that the pseudo-second-order model fits experimental data better than pseudo-first order because of its higher correlation coefficient value (R^2=0.9926). It means that the adsorption process is taking place by chemisorption which involves sharing electron between metal ion and surface of the adsorbent (El Qada et al., 2006). All kinetic constants for adsorption of Pb (II) ions by activated carbon made from kiwi peel at the initial concentration of 500 µg.L^{-1}, are summarized in Table 2.
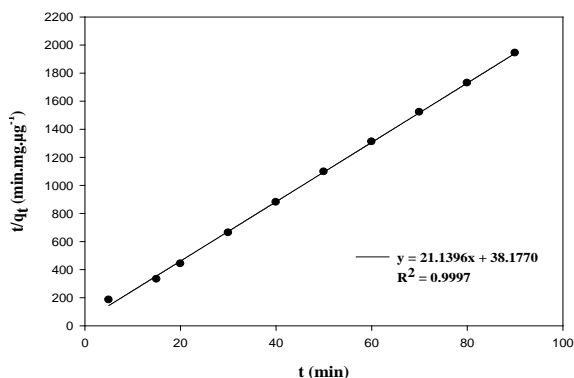
Fig. 8. Pseudo-second order model for adsorption of Pb (II) at 20°C, C_0=500μg.L^{-1}, pH=6

Table 2. Kinetic model parameters for Pb (II) adsorption on adsorbent

Kinetic model	Parameters	Value
Pseudo-first-order	k_1 (g/mg.min)	0.0449
	$q_{e,cal}$ (mg/g)	104.71
	R^2	0.9785
Pseudo-second-order	k_2 (g/mg.min)	0.0004
	$q_{e,cal}$ (mg/g)	95.23
	R^2	0.9926

3.4. Adsorption isotherms

Adsorption isotherm models are commonly used to represent the equilibrium adsorptive behavior and investigate its mechanisms (Wang et al., 2010). In this study, to find a relationship between the amounts of Pb (II) adsorbed onto the adsorbent and its equilibrium concentration, Langmuir, Freundlich and Temkin isotherm models were used.

The Langmuir isotherm (Langmuir, 1918), assumes that each site can hold one molecule of adsorbate. It means that adsorption occurs at equivalent sites of the adsorbent. The linear form of Langmuir isotherm model is given by Eq. (5):

$$\frac{C_e}{q_e} = \frac{1}{q_{max}K_L} + \frac{1}{q_{max}}C_e \qquad (5)$$

where C_e is the equilibrium concentration (μg.L^{-1}), K_L is the Langmuir adsorption constant which is related to the energy of adsorption and q_{max} (μg.g^{-1}) is the maximum adsorption capacity under experimental condition.

The Freundlich isotherm assumes that adsorption occurs on heterogeneous surface (Ng et al., 2002). It can be linearly expressed by Eq. (6):

$$logq_e = logK_F + \frac{1}{n}logC_e \qquad (6)$$

where K_F is adsorption capacity of the adsorbent (μg.g^{-1} (L/μg)$^{-1/n}$) and n represents bond energies between metal ion and the adsorbent.

These constants can be determined from the plot of log(q_e) against log(C_e). Temkin and Pyzhev (Temkin and Pyzhev, 1940) proposed that because of

the adsorbate-adsorbent interaction, the heat of adsorption of all molecules in the layer will reduce with the coverage linearly. Temkin isotherm is described as (Eq. 7):

$$q_e = BlnK_T + BlnC_e \qquad (7)$$

where: K_T (L.μg^{-1}) is equilibrium binding constant that corresponds to maximum binding energy. $B=RT/b$ indicates the heat of adsorption, R is universal gas constant (8.314 J.mol^{-1}), T is absolute temperature (K), and $1/b$ represents the adsorption potential of adsorbent. Both constants can be determined from plot of q_e against C_e (Hameed and Ahmad, 2009).
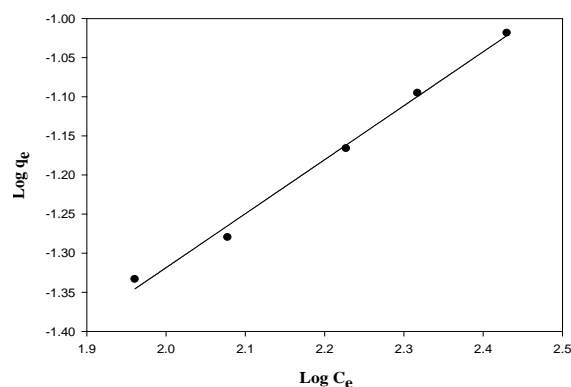

Fig. 9. Freundlich adsorption isotherm model for Pb (II) on activated carbon from kiwi peel; at 20°C, C_0=500μg.L^{-1}, pH=6

The equilibrium adsorption studies were carried out at different initial concentrations; 500, 600, 700, 800 and 1000 μg.L^{-1}. The isotherm parameters for adsorption of Pb (II) were calculated and given in Table 3. Freundlich isotherm model provides best fit with best correlation coefficient (R^2=0.9937) which indicates that the adsorption process involves multi-layer adsorption.

Table 3. Equilibrium parameters for adsorption of Pb (II)

Equilibrium model	Parameter	Value
Langmuir isotherm	K_L	0.0031
	q_{max}	0.1984
	R^2	0.9809
Freundlich isotherm	K_F	0.9928
	n	1.44
	R^2	0.9937
Temkin	K_T	0.5548
	B	0.0464
	R^2	0.9763

4. Conclusion

The present study showed that activated carbon made from kiwi peel is suitable for removing Pb (II) from aqueous solution. The effect of solution pH, adsorbent dosage, contact time, initial concentration and shaking rate wereinvestigated. The synthesized activated carbon was categorized as a mesoporous

material with surface area of 306.17 m^2.g^{-1}. SEM micrograph demonstrated numerous pores with different shapes and sizes. FTIR spectrum also confirmed the presence of some functional groups like hydroxyl groups or phosphoric compounds. It was observed that the adsorption of Pb (II) is pH dependent; the optimum pH was found to be 6.

The obtained results showed that shaking rate had insignificant effect on adsorption of Pb (II).The comparative study showed that kiwi peel-based activated carbon had the same potential as the commercial activated carbon in omitting Pb (II) from aqueous media. The kinetic study showed that the pseudo-second-order model had a better correlation coefficient (0.9997) than pseudo-first-order. This meant that chemisorption was dominant process.

In order to investigate the equilibrium data, Langmuir, Freundlich and Temkin isotherm models were used. The results showed that Freundlich isotherm fittedbetter the adsorption equilibrium data than the other models. It means that it described the division of the molecules between the solution and adsorbent better as opposed to the other models.

The results showed that low-cost precursors which are worthless, can be used for the development of effective adsorbents for wastewater treatment process especially, removing Pb (II) ions from aqueous systems for environmental protection purposes.

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