

A comparative study of raw and treated peach stones for the adsorption of hexavalent chromium: characterization, kinetic and thermodynamic modelling

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ABSTRACT/RESUME

Abstract : In the last years, much attention has been focused on the use of low-cost adsorbents for the removal of heavy metals from wastewater. The present study consists of comparing the adsorption of hexavalent chromium in aqueous solutions by two adsorbents: raw peach stones (RPS) and peach stones activated carbon (PSAC). The characterization of the adsorbents was done by using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Comparative study was carried out using adsorption parameters such as contact time, adsorbent dose and solution pH in a batch system. Results show that pH is key parameter for adsorption optimization. The time necessary to attain adsorption equilibrium was found 120 min for PSAC in the pH range 2-5.6 and 240 min for RPS at pH 2 with maximum removal percentage greater than 97% for both adsorbents. The adsorption process followed the pseudo-second order kinetics. Thermodynamic parameters revealed the spontaneous and endothermic nature of the adsorption process for both adsorbents. Investigations carried out proved that RPS and PSAC are good potential adsorbents for the treatment of toxic hexavalent chromium.

I. Introduction

The industrial discharges without any appropriate treatment containing heavy metals represents a significant and long-term environmental hazard [1]. Heavy metals have great harm to environment because they accumulate in creatures and plants and enter the human body through food chain [2]. Chromium is one of the most highly toxic heavy metal generated from many industrial processes, such as leather tanning, battery, pharmacy, electroplating, metallurgy, textile and catalyst synthesis [3,4]. Chromium exists in two oxidation states, hexavalent Cr (VI), and trivalent Cr(III) and the toxicity of chromium is closely related to the valence state [5]. Both have a significant influence on human health: Cr(III) is an essential ion, whereas Cr(VI) compounds are highly toxic, mutagenic and carcinogenic [6]. Hexavalent chromium is 100 times more toxic than trivalent chromium [7]. Therefore,

due to its high toxicity it is necessary to reduce Cr(VI) to acceptable levels before discharging it into the environments. According to the guidelines recommended by the World Health Organization (WHO), the maximum permissible limit for Cr(VI) in inland water is 0.1 mg/L and 0.05 mg/L for potable water [8]. Several treatment technologies have been used to reduce the Cr (VI) concentration of the industrial wastewater to the permissible level, such as membrane separation, chemical precipitation, ion exchange, reduction, electrochemical precipitation, solvent extraction, electrodialysis and adsorption [9,10]. However, high energy and chemical requirement, incomplete removal, generation of toxic sludge are the limiting factors of these treatment procedures [11]. The adsorption is recognized as a simple, inexpensive, and convenient process; it is considering an interesting alternative to operate mainly using economical and eco-friendly

biomaterials [12]. Adsorption process is used in a variety of important industrial applications and now it is increasingly used on large scale as an economical and efficient separation technique for metal ion removal from wastewater. Agricultural by-products exist in large amounts, which represent consequently a solid pollutant to the environment. In recent years, special attention has been focused towards valorization of these wastes for their uses in adsorption treatment. These wastes can be used raw [13] or treated (physically or chemically) [14]. This study deals with the biosorption ability of raw and treated peach stones for the removal of chromium from synthetic aqueous solutions.

II. Experimental

II.1. Preparation of adsorbents from peach stones

The adsorbents used in this study were prepared from agricultural waste biomass: peach stones. The collected raw peach stones, were first washed several times with tap water to remove the adhering impurities followed by distilled water, after drying in an oven at 110°C for 24 h, they were crushed and sieved to desired particle size (1-2mm). A portion of the resulting material obtained was used in the raw form without any physical or chemical treatment was named as RPS. The remaining portion of peach stones grains was subjected to carbonization in "Nabertherm" muffle furnace at 600°C for 40 min and the obtained carbonizate was submitted to a chemical activation with phosphoric acid (H₃PO₄) in a 1:3 (w/w) impregnation ratio. After filtration, the sample was cooled to room temperature and washed several times with hot and cold distilled water respectively, in order to remove the remaining phosphoric acid until a neutral pH. The washed sample was then dried at 110°C for 24 h and sieved to desired particle size (0.315 and 1mm) was named as PSAC and stored in a desiccator for use in adsorption studies.

II.2. Characterization of the obtained adsorbents

The prepared adsorbents (RPS and PSAC) were characterized using various techniques. The functional groups were analysed using Fourier transform infrared spectroscopy (Bruker ALPHA) at wavelengths in the range 400–4000 cm⁻¹. The morphology and elemental composition of the adsorbent were determined by scanning electron microscopy (SEM) (Quanta 650).

II.3. Batch Adsorption Experiments

The adsorbents prepared from peach stones were tested for the removal of Cr(VI) from aqueous solution in batch system. The experiments were performed in a series of 100 mL beakers containing 50 ml of the 30 mg/L Cr(VI) solution with a certain

mass of adsorbent at a constant agitation speed of 200 rpm. The effect of various process parameters such as pH, adsorbent dose and contact time on the percentage removal of Cr(VI) was studied.

Synthetic chromium solution was prepared by dissolving potassium dichromate in distilled water, after each experiment of adsorption, the solution was filtered and the residual concentration of Cr(VI) was determined by using spectrophotometer (UV-1800 SHIMADZU) at λ_{max} 540 nm after complexing with 1, 5-diphenylcarbazide in acidic medium [15]. The removal percentage (R %) and the biosorption capacity q_e (mg/g) of Cr(VI) by RPS and PSAC were calculated using the following equations:

$$R(\%) = \frac{C_0 - C_t}{C_0} * 100 \quad (1)$$

Where C₀ (mg/L) and C_t (mg/L) represent the concentrations initially and at time t respectively.

$$q_e = \frac{C_0 - C_e}{m} * V \quad (2)$$

Where V(L) is the solution volume, m(g) represents the mass of adsorbent, C₀ (mg/L) and C_e (mg/L) are the concentrations of Cr(VI) at initial and equilibrium in the solution, respectively.

III. Results and discussion

III.1. Characterization of the adsorbents

III.1.1. Analysis by SEM

The SEM images of the surface structure for raw and modified peach stones at different magnified times (Figure 1) shown significant differences in the surface morphology. It is clear from the SEM image that the external surfaces of both samples were rough and contained pores of different size and shapes. The representative SEM figure 1 (a,b,c) show that the external surface of RPS presents irregular porous nature structure with small pores and cavities. This porosity was formed during the oven-heating step in the preparation of the raw adsorbent [16]. The morphology of the PSAC presented in figure 1(d,e,f) show a highly developed porosity over the entire surface of the prepared adsorbent with homogenous structure contained deep pores and many large cavities. In addition, there may be also the presence of some fine particles attached to the activated carbon. These particles can be attributed to both a reminiscence of vegetable origin of adsorbent and impurities formed during its preparation [17]. The creation of more pores and cavities on the surface of PSAC may be due to the modification treatments by H₃PO₄.

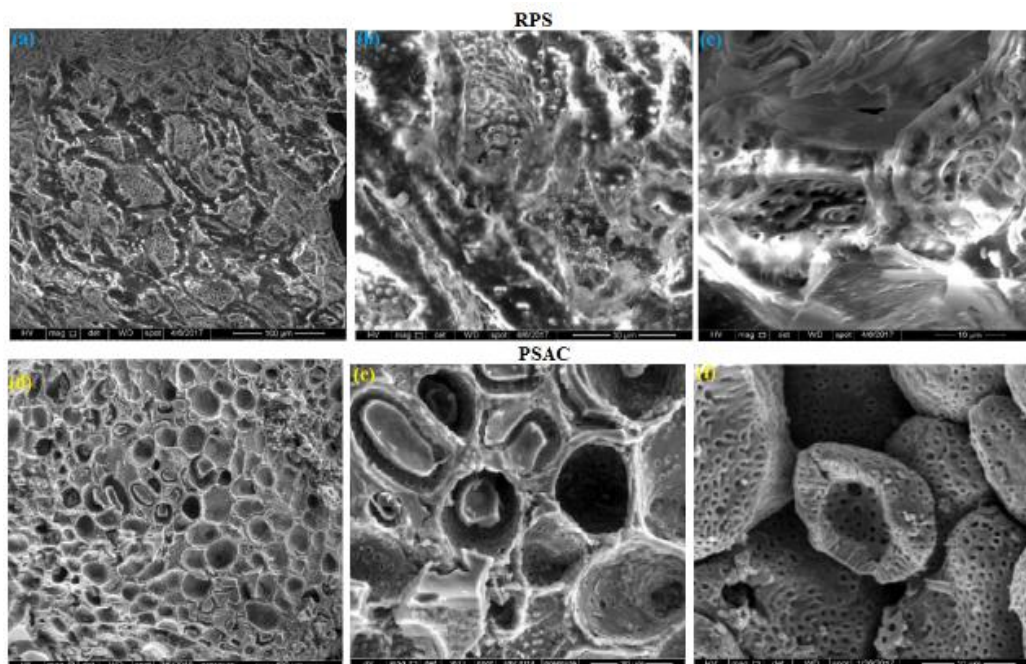


Figure 1. SEM images of (a, b, c) RPS and (d, e, f) PSAC.

III.1. 2. FTIR analysis

The FTIR spectrum of the raw and prepared activated carbon from peach stones are illustrated in figure 2 and the positions with assignments of bands are presented in table 1. The results are indicated that there are a number of important functional groups on the surface of the both precursors (RPS and PSAC). As can be seen from figure 2 some changes on FTIR spectrum of PSAC, where three bands located at

1508, 1420 and 1047 cm^{-1} have disappeared during the carbonization at high temperature and the chemical treatment. Moreover, FTIR spectra of RPS and PSAC show reduction of intensity of bands (3440, 1735, 1633, 1261 cm^{-1}) in RPS to (3418, 1703, 1600, 1237 cm^{-1}) in PSAC. In general, the shift in the FTIR spectra, reduction and disappearance of the peaks of each functional group are due to the fact of carbonization temperature and chemical treatment of peach stones waste.

Table 1. FTIR peak comparison between raw peach stones (RPS) and treated peach stones (PSAC)

Band position cm^{-1}		Assignment	References
RPS	PSAC		
3440	3418	Carboxylic/OH stretching and N-H stretching	[18,10]
2925	2924	Aliphatic C-H stretching	[19,20]
2854	2854	-CH stretching vibration in -CH and -CH ₂	[10,21]
1735	1703	C=O stretching vibration of carboxylic groups	[22,23]
1633	1600	C=C stretching vibration of aromatic ring structures	[23,19,24]
1508	/	C-C/ C=C stretching vibration of aromatic ring	[25]
1457	1458	C-C/ C=C stretching vibration of aromatic ring	[25]
1420	/	C-C stretching of aromatic ring	[26]
1383	1383	Symmetric bending of OCH ₃ or -CH ₃ symmetrical deformation mode (scissoring) in amide group	[18,10]
1261	1237	C-O stretching	[27,19]

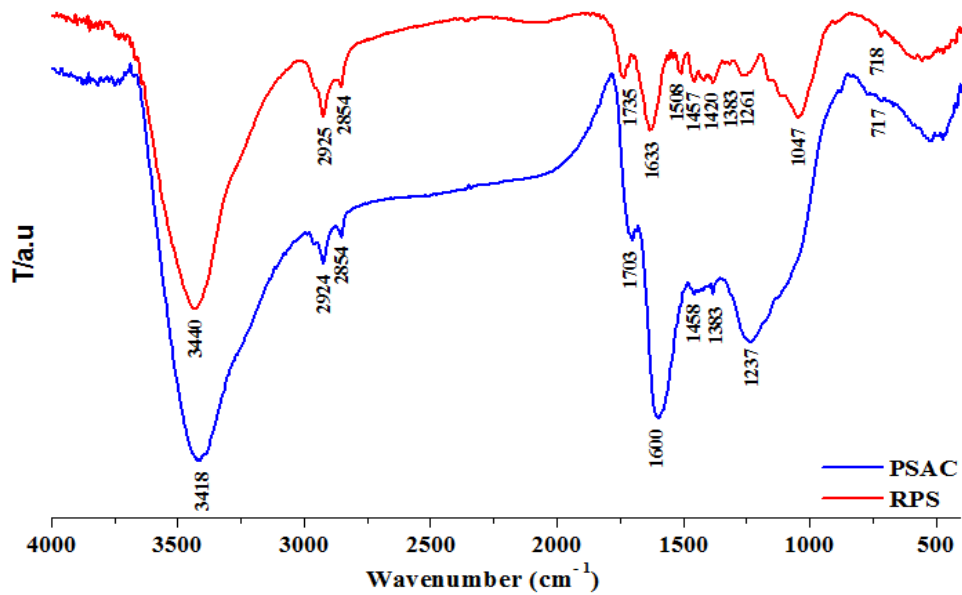


Figure 2. FTIR spectra of raw peach stones (RPS) and activated carbon (PSAC).

III.2. Biosorption process

III.2.1. Effect of adsorbent dose on Cr (VI) removal

The effect of biosorbent dose on Cr(VI) removal onto RPS and PSAC is shown in Figure 3. It was investigated using 1 to 10 g/L adsorbent dose at initial Cr(VI) concentration of 30 mg/L. Results show that the removal of Cr(VI) increases with increase in adsorbent dosage for both RPS and PSAC. The Cr(VI) percentage removal by RPS and PSAC increased from 37% to 97% and 13% to 98% by increasing the adsorbent dosage from 1 to 4g/L and 1 to 6g/L respectively. The increase in Cr(VI) percentage removal at higher adsorbent dose may be due to the fact that higher surface area is available for the adsorption process [28]. The optimal adsorbent doses of 4 g/L(RPS) and 6 g/L(PSAC) were selected for further adsorption experiments in this study.

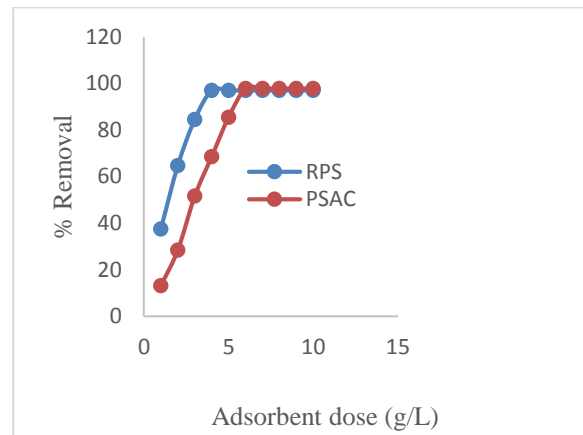


Figure 3. Effect of RPS and PSAC dose on percentage removal of Cr (VI).

III.2.2. Effect of initial pH solution on Cr (VI) adsorption

The pH of the solution is one of the most important controlling parameter in the chromium biosorption process [29]. It controls the surface properties of the biosorbent and adsorbate speciation [30]. The adsorption of Cr (VI) ions on RPS and PSAC was investigated at pH ranging from 2 to 11. The plot of percentage removal of Cr(VI) represented in figure

4 shown that the Cr(VI) adsorption on RPS and PSAC was strongly dependent on the initial solution pH. As can be seen from figure 4, the maximum percentage chromium removal for the RPS was 97 % at pH 2 and 98% for the PSAC at pH ranging from 2-5.6. Also, it was observed a drastic decrease in Cr(VI) removal at pH >2 for RPS and pH>5.6 for PSAC. At a lower pH between 2 and 6, the dominant form of Cr(VI) is HCrO_4^- and $\text{Cr}_2\text{O}_7^{2-}$ and under alkaline conditions (pH >8) it exists predominantly as chromate anion. At lower pH, the adsorbent surface is positively charged that results in strong electrostatic attraction between positively charged

adsorbent surface and negatively charged chromate ions. At higher pH the surface of adsorbent becomes negatively charged and also there is abundance of hydroxyl ions in aqueous solution [31,5] resulting in the repulsive force between Cr(VI) anions and negatively charged biosorbent surface. Additionally, higher pH decreasing the removal may be explained by the competitive adsorption between chromate and hydroxyl ions [32]. Therefore, the optimum pH values for the adsorption of Cr(VI) onto RPS and PSAC were 2 and 5.6 (free pH solution) respectively.

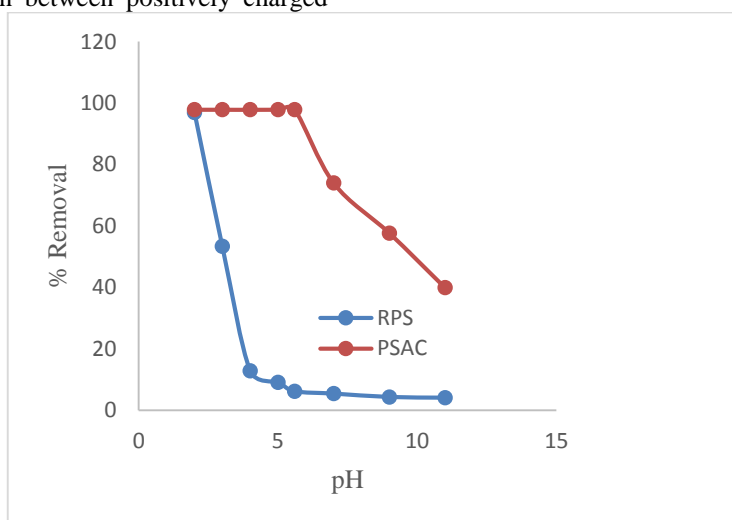


Figure 4. Effect of pH on the percentage removal of Cr(VI) by RPS and PSAC.

III.2.3. Effect of contact time on Cr(VI) removal

The effect of contact time on Cr(VI) removal on RPS and PSAC was investigated at different time intervals up to 480 min at an initial Cr(VI) concentration of 30 mg/L and using the other parameter values mentioned previously. As shown in figure 5, the percentage removal of Cr(VI) onto both RPS and PSAC increased with an increase in the contact time. The curves show that equilibrium was reached after approximately 120 min for peach stones activated carbon (PSAC) and after 240 min for raw peach stones (RPS).

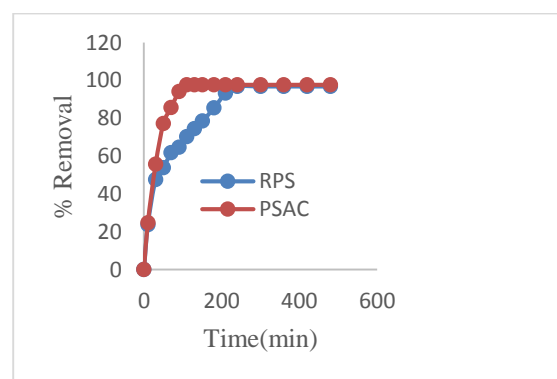


Figure 5. Effect of contact time on percentage removal of Cr(VI) by RPS and PSAC.

III.3. Kinetics of Cr(VI) adsorption

The kinetics of adsorption shows the relationship between rates of adsorption with its contact time. Several kinetic models can be used to explain it. In this work the adsorption kinetics of Cr(VI) on RPS

and PSAC were evaluated using pseudo first-order and pseudo second-order models. The equations

related to these kinetic models are given below, respectively:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

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

where q_e and q_t are the amount of Cr(VI) adsorbed (mg/g) at equilibrium and at time t (min) respectively, k_1 (min⁻¹) and k_2 (g/mg min) are the

kinetic rate constants for the pseudo-first order and pseudo-second-order .

The kinetic modeling of the experimental of Cr(VI) onto RPS and PSAC is shown in figure 6. All the obtained kinetic parameters calculated from linear plots and correlation coefficient (R^2) values were represented in table 2. Straight lines were achieved for both adsorbents based on the pseudo-first-order and pseudo-second-order models (Figure 7). The calculated values of adsorption capacities (q_e) showed good agreement with experimental values. On comparison of R^2 values, it can be concluded that the adsorption of Cr(VI) ions onto RPS and PSAC followed the pseudo second order kinetic model.

Table 2. Adsorption kinetics parameters of Cr(VI) on RPS and PSAC

Kinetic model	Pseudo-first order parameters				Pseudo-second order parameters		
Parameters	q_e (mg/g)	q_e (mg/g)	k_1 (min ⁻¹)	R^2	q_e (mg/g)	k_2 (g/mg.min)	R^2
RPS	7.27	6.778	0.0127	0.9236	8.4175	0.0022	0.9757
PSAC	4.843	5.604	0.037	0.9690	6.882	0.003	0.9949

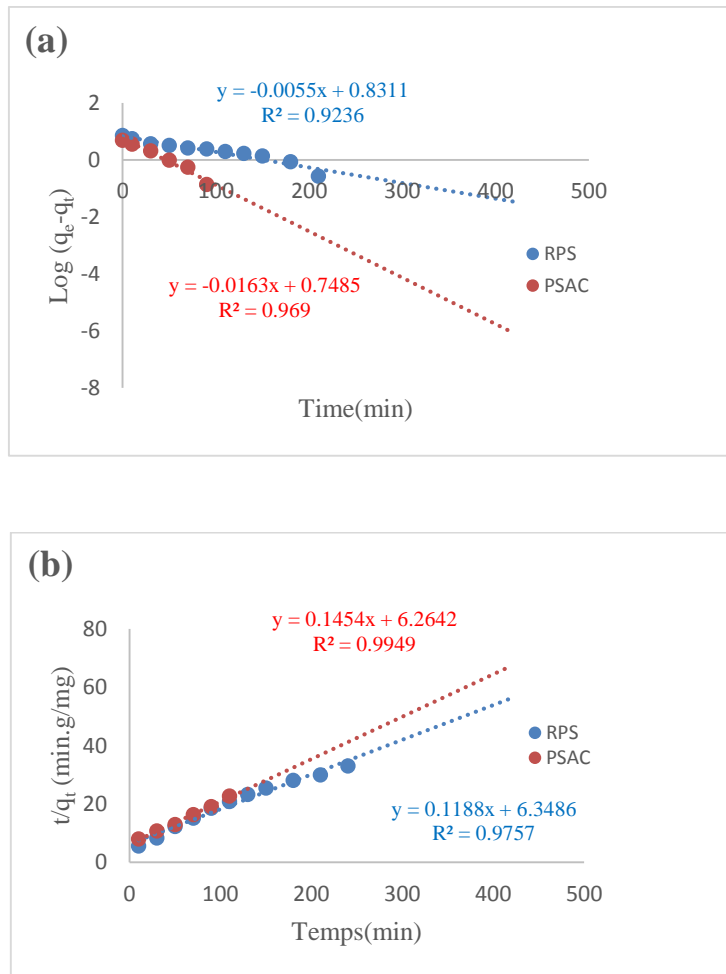


Figure 6. Pseudo first order (a), Pseudo second order (b) kinetics of RPS and PSAC.

III.4. Thermodynamic parameters of Cr(VI) adsorption

The thermodynamic parameters such as entropy change (ΔS), enthalpy change (ΔH) and Gibb's energy (ΔG) were determined for temperatures ranging from 283K to 313K to evaluate the feasibility and nature of Cr(VI) adsorption by adsorbent, using the following expressions:

$$\Delta G = -RT \ln K \quad (5)$$

$$\ln K = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (6)$$

where R (8.314 J/mol K) is the gas constant, T (K), absolute temperature and K is the equilibrium constant, which represents the ratio between the concentration of solute that is adsorbed and the concentration of solute remaining in solution, can be calculated using the expression:

$$K = \frac{C_0 - C_e}{C_e} \quad (7)$$

where C_0 and C_e are the initial and equilibrium concentrations (mg/L), respectively of Cr(VI).

The values of ΔH and ΔS were calculated from the plot of $\ln K$ vs $1/T$ as shown in figure 7. The values of ΔH , ΔS and ΔG for the biosorption of Cr(VI) onto RPS and PSAC at various temperatures are listed in table 3. The positive value of ΔH indicate the endothermic process of adsorption and that an increase in temperature favored the adsorption process. The negative values of ΔG show that the adsorption process is feasible and spontaneous. The ΔS value is positive, which suggested that the randomness increased at solid- solution interface during the adsorption of Cr(VI) on the both adsorbents (RPS and PSAC).

Table 3. Thermodynamic parameters for the adsorption of Cr(VI) by RPS and PSAC

Adsorbent	T(K)	C_e (mg/L)	q_e (mg/L)	$\ln K$	ΔG (kJmol ⁻¹)	ΔH (kJmol ⁻¹)	ΔS (kJmol ⁻¹ K ⁻¹)
RPS	283	7.155	5.711	1.161	-1.793	97.257	0.350
	293	2.176	6.956	2.548	-5.293		
	303	0.921	7.270	3.452	-8.793		
	313	0.255	7.436	4.759	-12.293		
PSAC	283	4.477	4.254	1.741	-2.878	107.492	0.39
	293	2.038	4.660	2.619	-6.778		
	303	0.376	4.937	4.367	-10.678		
	313	0.126	4.979	5.468	-14.578		

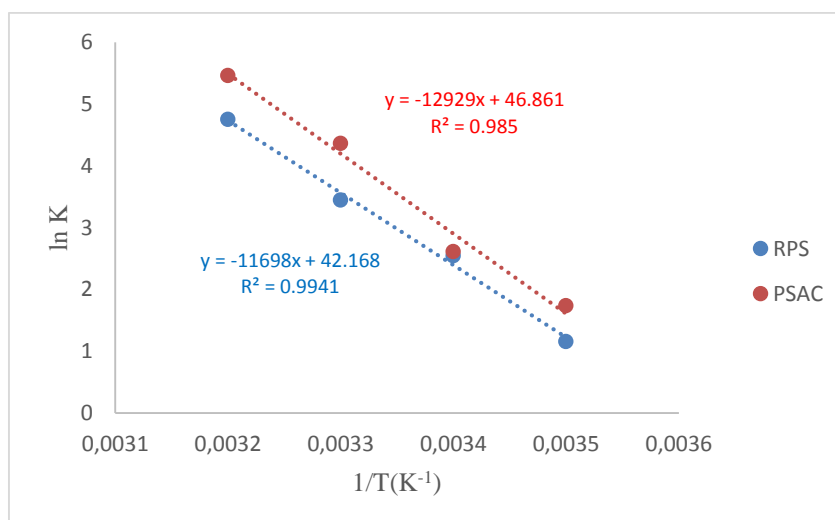


Figure 7. Plot of $\ln K$ versus $1/T$ for Cr(VI) adsorption on RPS and PSAC .

IV. Conclusion

This study discussed the adsorption of hexavalent chromium ions by raw and modified peach stones. The characterization results showed a well-developed texture characteristics of the PSAC after carbonization, and acid treatment that proved that the temperatures of carbonization and activation were favorable for the opening of micropores and mesopores. The FTIR analyses showed the presence of significantly functional groups for both adsorbents. Solution pH played an important role on Cr(VI) percentage removal for the used adsorbents. The results show that Cr(VI) adsorption by RPS was favorable at low pH 2 whereas for PSAC the adsorption was favored in the pH range of 2-5.6. The time necessary to attain adsorption equilibrium was found 120 min for PSAC and 240 min for RPS with removal efficiency greater than 97%. Adsorption kinetics followed second order model. Thermodynamic study showed that the adsorption is spontaneous and endothermic for both adsorbents. Further, it can be concluded that the abundant low cost agriculture biomass, which contain high amounts of natural polymers such as cellulose, hemicellulose, and lignin, are interesting precursors for the adsorption process. Additionally, the present work demonstrated that RPS and PSAC can be effectively applied for the removal of toxic Cr(VI) from waste water.

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