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Paracetamol and amoxicillin adsorptive removal from aqueous solution using phosphoric acid activated-carbon

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Abstract: Charcoal-based materials have attracted much attention for the removal of pharmaceutical agents. The charcoal-based carbon materials have green synthetic routes, high surface area, numerous active site with active functional groups available for physico-chemical interactions with adsorbate for surface-adsorptive removal of toxins. In this study, acid treated activated carbon was developed from the peach seeds using thermal pyrolysis approach. Phosphoric acid activated carbon (PAC) was further modified by HNO_3 and employed as an adsorbent for the removal of amoxicillin and paracetamol and process variables were optimized for enhanced removal of amoxicillin and paracetamol. The adsorption of pharmaceutical agents was significantly affected by temperature, pH and reaction time. The amoxicillin and paracetamol sorption process onto PCA followed a pseudo second order kinetics and Langmuir isotherm model with a maximum removal capacity of 51.8 mg/g and 51.1 mg/g, respectively. The results revealed that acid activated carbon has promising

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efficiency for the removal of amoxicillin and paracetamol from aqueous medium and peach seeds derived PCA could be employed for the removal of these pharmaceutical agents from effluents and PAC is also extendable for the removal of other drugs from pharmaceutical wastewater streams.

Keywords: activated carbon; adsorption; environmental pollution; kinetics; pharmaceutical agents.

1 Introduction

Widespread concern regarding the abundance of pharmaceuticals in wastewater streams has largely increased since last two decades. The existing treatment technologies are not considered appropriate for removal of micro-pollutants as these are not designed to handle such pollutants [1–5]. Freshwater is vital to initiate and maintain the food chains at various levels. Natural processes i.e., photosynthesis, evaporation and unnatural processes, i.e. textile-processing, leather tanneries [6], paints and pigment synthesis, cement manufacturing and pharmaceutical productions are the main consumer of fresh water on large-scale. Eco-toxicity, mainly from water resources on living beings, has been a principal concern for the last three decades [7–10]. Pharmaceutical products are playing a vital role in the treatment of various diseases to maintain a healthy and quality life across the globe since the revolution of medicinal chemistry. Due to stressed lifestyle, lack of sports and exercise facilities in under-developing countries, number of diseases are multiplying day by day. Consequently, the pharmaceutical-industrial expansion has been achieved to provide the excellent possible life saving-drugs to serve humanity [11, 12]. Such life-saving drugs can be classified into various classes for instance, antimalarial, antiseptics, analgesics [13], antipyretics and antibiotics [14] etc. Amongst them, antipyretics and antibiotics are the drugs used to reduce fever, a common disease almost everyone could suffer easily prior to region, weather and age group. Aspirin, Panadol, paracetamol (PCT) and amoxicillin (AMX) are the antipyretics available in the first-aid box of every house, office, school, college and university. The ever-mounting demands of these medicines need to be satisfied only with large-scale manufacturing; resultantly the industrial wastewater contaminated with enormous concentration of these drugs will be discharged to nearby lakes [15].

The economical and facile removal of toxic effluents from industrial wastewater is highly demanded to ensure water quality for humans and plants. There are several methods to remove effluents from industrial water i.e. chemical method (nano-catalysts), and biological method (bio-adsorbents). Bio-sorption by expanding biomass is one of the utmost low-cost, facile and highly effective routes to degrade

industrial effluents [16, 17]. Previously, this technique was extensively applied for catalytic dye-degradation to remove organic dyes from industrial wastewater. It is also equally acceptable for the degradation of the drug due to its unique features, i.e., hierarchical porous topography, extended surface area, universal functional nature and excellent adsorption capacity [18–21].

Green and facile approaches to utilize the plant based adsorbent have played a vital role in nano-adsorbents; because plants have different origins and ecological evolution. This ecological evolution provides the varying composition with wide range of functionalities [22–24]. All the parts of plants are being utilized as adsorbents like leaves, stems and seeds. Plant seeds have extended surface area, smaller grain size and high quantum yield comparatively. Plants seeds-based adsorbents have shown a promising future for effluents removal from domestic and industrial wastewater. Peach seeds are a waste product and produced of 48,284 tons in Northern territory of Pakistan customary harvest and possess the regions of Quetta, Kalat, Peshawar, Swat valley and certain places of Kohistan slopes are the basic developing regions of peach., which have potential to of a good adsorbent by minor treatments. Rafatullah et al. has reported peach seeds derived activated carbon as an efficient and cost-effective adsorbent for degradation of methylene blue dye [25]. The seeds derived activated carbon has excellent ability to adsorbed pharmaceutical drugs due to their variant surface functionalities and surface defects created by the thermolysis of biomass.

Herein, we are reporting a cost-effective, facile and proficient biomass-derived adsorbent peach seeds activated carbon. The carbon obtained after pyrolysis at 430 °C was activated with phosphoric acid (PAC) and finally, treated with nitric acid to remove oxygenated functionalities from the activated surface. The prepared adsorbent was used for the removal of amoxicillin and paracetamol from aqueous medium as a function of various process variables and data this obtained was computed using isotherms and kinetics models.

2 Materials and methods

2.1 Materials

Standard solutions of phosphoric acid (12 M), nitric acid (6 M) and sodium hydroxide (ICI Chemicals Pakistan) were purchased for Sigma-Aldrich. Pharmaceutical agents like amoxicillin (Abbot Industries Pakistan) and paracetamol (Glaxo Smith Kline Internationals Pakistan) were used as adsorbate. All the chemicals used in the current work of analytical grades and utilized as without further processing.

2.2 Preparation of activated carbon

Peach seeds were extracted and washed 5 times, then crushed, dried, then this dried product was screened and sieved of 0.5–0.589 mm size. Precursor material (60 g) will be impregnated with phosphoric acid in round bottom flask reactor at 85 °C for 6 h, filtered under vacuum to remove excess of phosphoric acid, finally calcined at 430 °C for 4 h. Then Finally, the calcined product was washed with ultrapure water to control the pH of 6.5. lastly solid was dried in an oven for 24 h at 110 °C. The solid charcoal was used as phosphoric acid activated carbon (PAC); activated carbon modified with concentrated HNO_3 to remove the oxygenated functional groups from activated carbon (Figure 1).

2.3 Batch adsorption study

The adsorption kinetics were executed by dispersing 300 mg of PAC in a round bottom flask with 100–600 mg/L of pharmaceuticals containing water, with an optimized pH of 6.5–7 under continuous exposure of ultra-sonication at 30 °C temperature. The amount of adsorbed PCs was determined by using the following Eq. (1),

$$q_t = \left[\frac{(C_o - C_e)V}{m}\right] \tag{1}$$

Here, q_t is the amount adsorbed by PAC at time 't' (mg/g), C_o and C_e are the initial and equilibrium concentrations of PCs in the reaction mixture, 'V' is the volume PCs and 'm' is the mass of PAC, respectively [26–28].

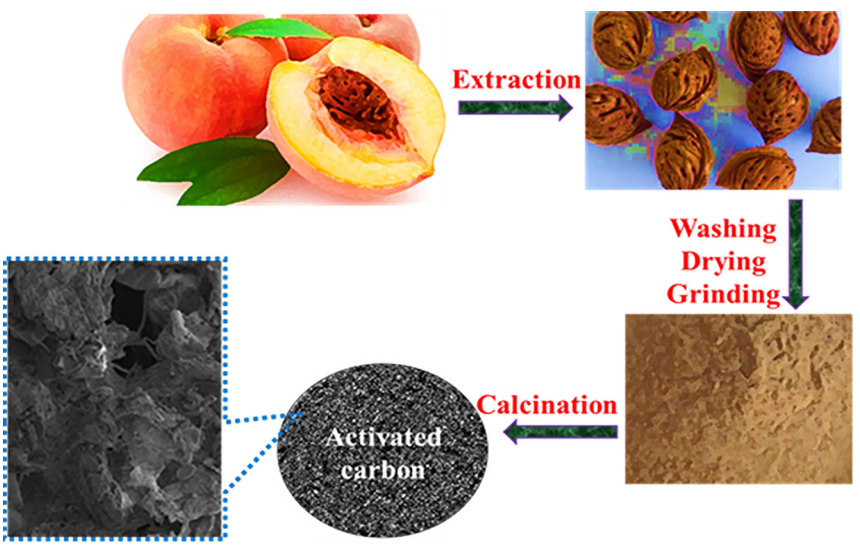

Figure 1: Layout of step-wise processing and preparation of activated carbon from peach seeds.

2.4 Characterization

Functional group affirmation of PAC was analyses using Fourier Transformation Infrared Spectroscopy (FTIR) by utilizing ATR powdered method with Bruker Alpha-2 with frequency ranges from 4000–600 cm^{-1}. Elemental composition of bio-sorbent and morphology of PAC was inspected by (SEM ZEISS SUPRA 55) scanning electron microscopy (SEM). Adsorption trends of amoxicillin and paracetamol were quantitatively monitored a UV-Visible spectrophotometer (Shimadzu UV-250 IPC). All the calculations have been made using MS excel and equations were defined using math type software.

3 Results and discussion

3.1 FT-IR analysis

Figure 2a depicts the absorption peaks of the loaded PAC with paracetamol in the FT-IR spectra. The intensities of peaks 3400-3200 cm^{-1} and 2925-2850 cm^{-1} –OH and

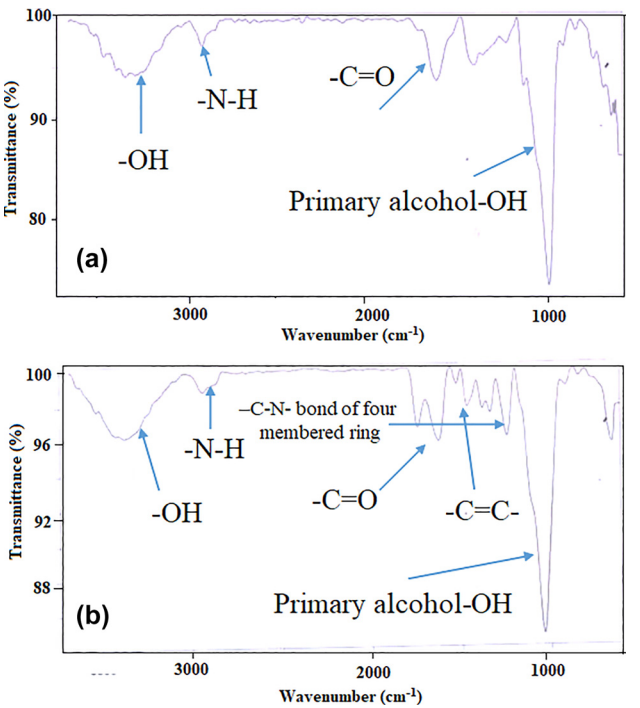

Figure 2: FTIR spectra of PAC loaded with (a) paracetamol and (b) amoxicillin.

–N-H stretching's of the paracetamol. The stretching's of a carbonyl with intramolecular hydrogen bonding's was observed at 1685-1665 cm^{-1}. Only a single intense and broad peak was present in the 1085-1050 cm^{-1} belongs to primary –OH group of the paracetamol. All the peaks present confirms the presence of the paracetamol adsorption at PAC surface.

Figure 2b depicts the absorption peaks of the loaded PAC with amoxicillin in the FT-IR spectra. The intensities of peaks 3400-3200 cm^{-1} and 3000-2850 cm^{-1} –OH and –N-H stretching's of the amoxicillin. The stretching's of a carbonyl with intramolecular hydrogen bonding's was observed at 1685-1665 cm^{-1}. The peak at 1650-1610 cm^{-1} belongs to the stretching of –C=C– of the aromatic ring. The peak at 1450 cm^{-1} affirms the presence of methyl groups. The stretching around 1250-1210 cm^{-1} belongs to –C–N– bond of four membered ring. Only a single intense and broad peak was present in the 1085-1050 cm^{-1} belongs to primary –OH group of the amoxicillin. All the peaks present confirms the presence of the amoxicillin adsorption at PAC surface.

3.2 Morphology and composition analysis

The surface features and morphology of the as-prepared PAC adsorbent were determined by scanning electron microscopy (Figure 3a–d). Adsorption sites are frequently available as facilitated by the macro-porous structure of PAC envisioned in Figure 3a. PAC samples loaded with pharmaceutical drugs have no porous active site; approximately maximum active sites were occupied with the adsorbent molecules (Figure 3b, c). Availability of some pores at the surface suggested that the equilibrium has been achieved, and maximum adsorption capacity of the PAC was attained. The pore size of unloaded and unloaded PAC sorbent were ~ 2 μm and 0.5–0.2 μm, respectively. The elemental composition of PAC includes carbon (42%), nitrogen (28%), calcium (10%), oxygen (6%), and a trace amount of sulfur, silicon, copper and magnesium. The oxygen was present in carboxylic acid because by-products of the combustion process were removed with nitric acid washing.

The carbon obtained after calcination at 450 °C and treatment with phosphoric acid owed macro-porous structure, with hierarchical channels. After performing adsorption studies, the macro-porous structure was converted to mesoporous structure occupied by the drugs. The size of amoxicillin and paracetamol was reported ~ to 5–20 μm and 90–200 μm respectively. The active-sites of the porous carbon was completely occupied as a result of adsorption the pore-size of carbon reduced [29, 30].

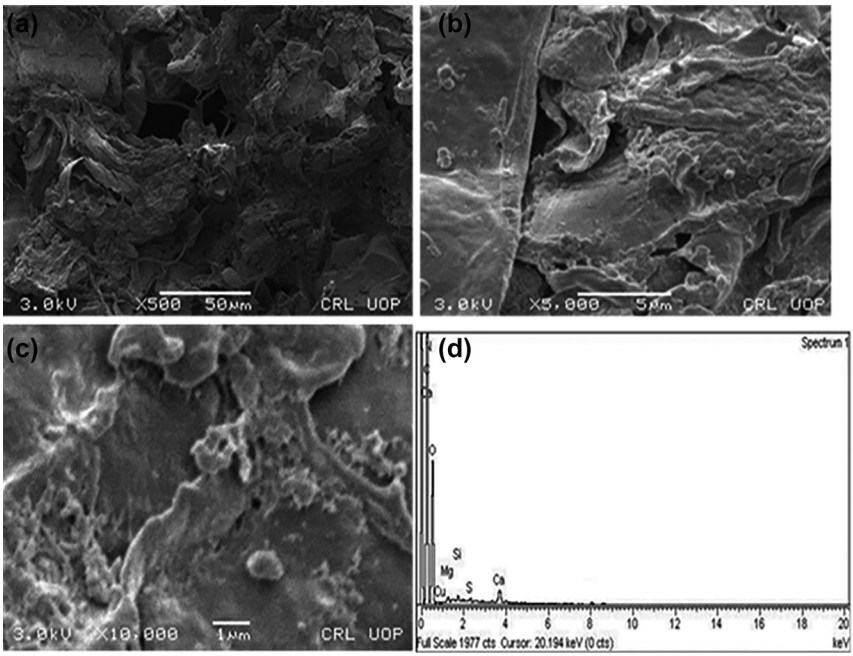

Figure 3: Macro-porous structure of PACs derived from peach seeds based bio-waste 1–50 µm (a–c) and EDX analysis of PAC (d).

3.3 Optimization of process variables

Adsorbent dosage impacts on exchange proficiency of PAC was investigated for the AMX and PCT. The adsorption was continued for one hour, PCs separately with an underlying gram of 300 mg/L. Mixing was completed at 30 °C temperature, 6.5–7 pH at 200 rpm for both PCs. At diverse adsorbent dosage ranges from 0.1–0.6 g, adsorption efficiency was estimated and outcomes (Figure 4). A pattern in removal efficiency with augmentation in measure of adsorbent dosage demonstrate that adsorption increased linearly with adsorbent mass while at 0.5 g–0.6 g plot became constant indicated that equilibrium was established. To check how the underlying centralization of pharmaceutical compounds concentration impacts (PCCI), adsorption of AMX and PCT ranges from 100–600 mg/L with an interval of 100 mg/L. AC mass was increased 0.3 g, and mixing was achieved at room temperature, neutral pH and 200 rpm for 1 h to both PCs; impact of initial concentration of AMX and PCT outcomes. Rising curves indicate that when the concentration of compounds is increased, there might be multi-layers' adsorption on the surface of PAC.

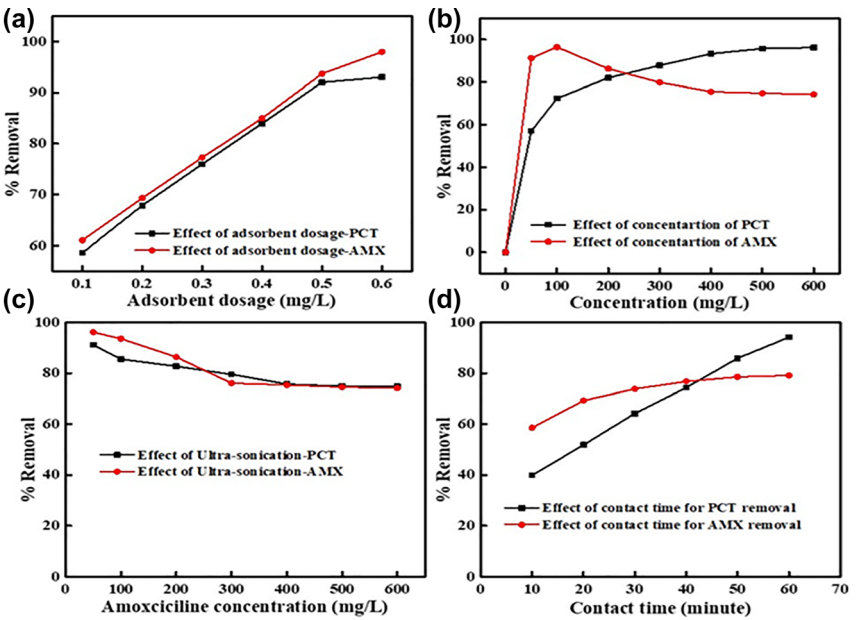

Figure 4: Factors effecting the removal efficiency of the PACs (a) effect of adsorbent dose (b) effect of concentration of amoxicillin and paracetamol, (c) ultra-sonication effect and (d) effect of contact time on the adsoprtion.

To investigate the impacts of ultrasonic beams on adsorption of AMX and PCT, group analysis was completed for both PCs separately. The contact time for PCs with ultrasonic beams was kept steady for 5 min; both PCs were shifted from 100–600 mg/L with an interim of 100 mg/L with adsorbent portion 0.3 g at room temperature with neutral pH of both PCs (Figure 4c). Information acquired from Figure 4c clarified that the connection between adsorption of PCT and AMX, ultrasonic stirring contact time; plots indicated that the PCs are lowest % R maximum with increasing concentration decreased the removal efficiency. The impact of contact time on adsorption of AMX and PCT was monitored, cluster procedure of adsorption was done for 10, 20, 30, 40, 50 and 60 min for both PCs. The adsorbent mass was 0.3 g with beginning centralization of PC 300 mg L^{-1} blending at room temperature, 7 pH for both PCs at 200 rpm (Figure 4d). The pattern of the two diagrams demonstrates the connection between contact time and expulsion productivity for both PCs there is linear relationship between stirring time and removal % R efficiency.

Optimization of pH impacts on adsorption of AMX and PCT was determined at pH 2–14 for both PCs exclusively. The adsorbent mass was 0.3 g with an underlying PCC 300 mg/L mixing was carried at room temperature with mixing time 1-h at

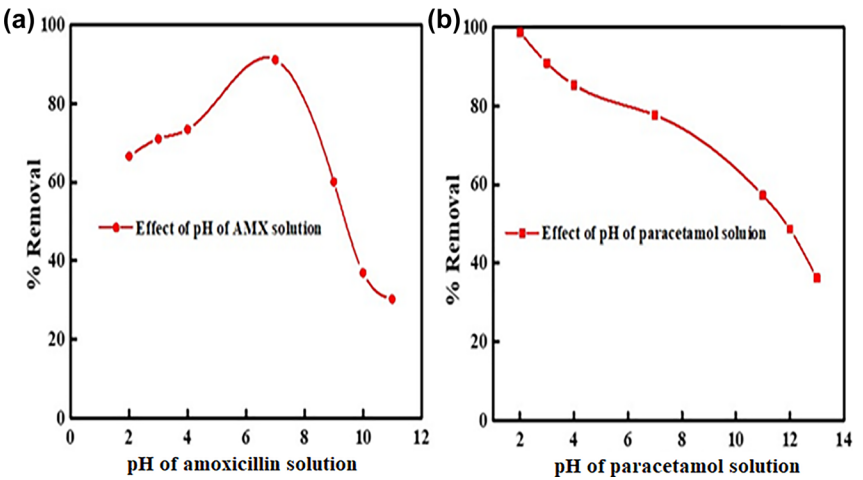

Figure 5: Effect of alkaline and acidic media on adsorption, (a) amoxicillin and (b) paracetamol.

200 rpm. The outcomes show the impact of pH on adsorption of AMX and PCT; demonstrates that % R of AMX is at neutral pH due to decomposition occurs in acidic as well in basic medium; % R of PCT is maximum in acidic medium and there is a decrease in efficiency basic and neutral pH (Figures 5a, b).

3.4 Adsorption modeling

Figure 6 exhibits the adsorption performance and 2-kinetic models of PAC for AMX and PCT with starting PCs concentration of 100 mg L^{-1} for 1 h at 30 °C having pH = 6.5–7. The equilibrium for these specific reactions was assessed after sixty minutes for AMX and PCT; PAC has shown excellent removal capacity 51.8, 51.1 mg/g for AMX and PCT, respectively. Approximately 200 mg L^{-1}, 300 mg L^{-1}, 400 mg L^{-1}, 500 mg L^{-1} and 600 mg L^{-1} were mixed at 200 rpm. The arrangements were separated, and their absorbance was estimated with a uv-noticeable spectrophotometer at determined wavelengths. The adsorption information got was relevant for Langmuir isotherm and Freundlich isotherm. Explained C_e/q_e designed against C_e for AMX and PCT individually. Isotherm of Langmuir was not obeyed by both PC detailed from out R^2 values for straight plots. The values of Langmuir constants, for example, K_L, R_L, q_{max} and R^2 for AMX and PCT orderly. The straight plot b/w log q_e and log C_e for both AMX and PCT individually demonstrated Figure 6 and Table 1, respectively. Linear plot for batch adsorption information with high co-efficient of relativity (R^2) PC obeyed by isotherm of Freundlich. Freundlich constants were determined from slope

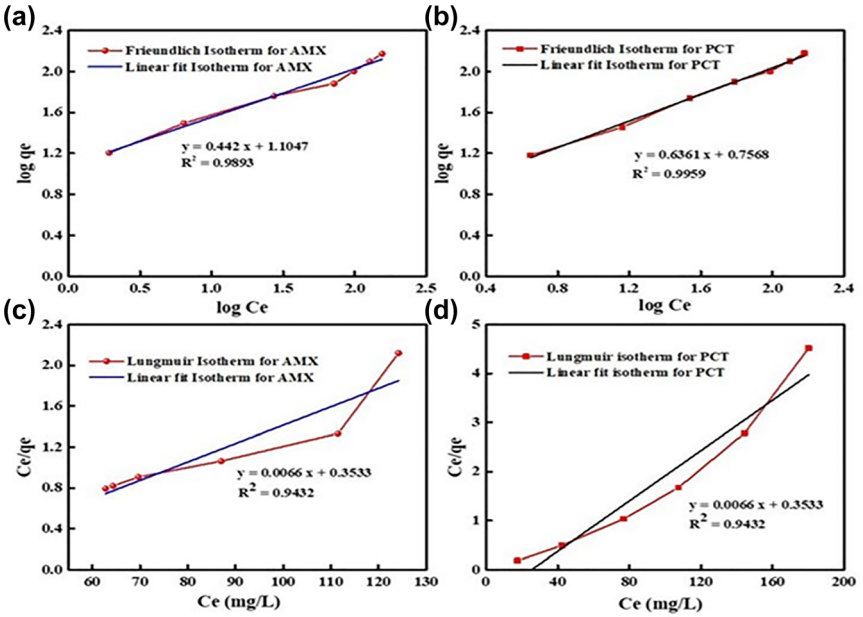

Figure 6: Freundlich isotherm for (a) amoxicillin removal and (b) paracetamol removal; Langmuir isotherm for (c) amoxicillin removal and (d) paracetamol removal.

Table 1: Langmuir and Freundlich parameters for sorption kinetics of PAC sorbent.

Samples	Langmuir Isotherm parameters			Freundlich Isotherm parameters		
	q_{max} (mg/g)	K_L (L/mg)	R^2	K_F (mg/g)	N	R^2
PAC (AMX)	51.8	0.0443	0.9432	12.7	2.26	0.9959
PAC (PCT)	51.1	0.0037	0.9432	5.7	1.57	0.9893

and intercept of linear plot. AMX and PCT values of Freundlich constants i.e., 1/n, adsorption intensity (n), adsorption capacity (K_f) and (R^2) co-efficient of relativity are given, respectively in Table 1.

3.5 Kinetics studies

Langergen, Morris and Weber initially used to calculate the rates of adsorption rates for kinetic models of Pseudo First and Second Orders. The plot of log (q_e-q)

against time respectively. The linear plot and high (R^2) co-efficient relativity i.e. 0.9963 confirmed the use of 2nd order kinetics for AMX. Figure 7 exhibited the plot between t/q_e and time. Linearity of chart and large value of R^2 = 0.9594 encourage the use of 2nd order kinetics also for PCT. The calculated value of PCT is very close to 2nd order kinetics than values of 1st order kinetics hence batch data for PCT best fitted 2nd order kinetics, respectively. The activated carbon derived from various routes always contains surface active groups due to the heteroatom doping caused by thermolysis process. Such doping primarily creates surface active groups like –OH, -COOH, -NH and may be the combination of all of the mentioned groups; such surface active results van da wall interactions or chemical bonds with the active groups of the pharmaceutical agents [31, 32].

Table 2 shows the comparison among the different bio-sorbents already employed for adsorption of these drugs and the present work. It clearly indicates that adsorbent used in this study has shown comparable adsorption of these drugs.

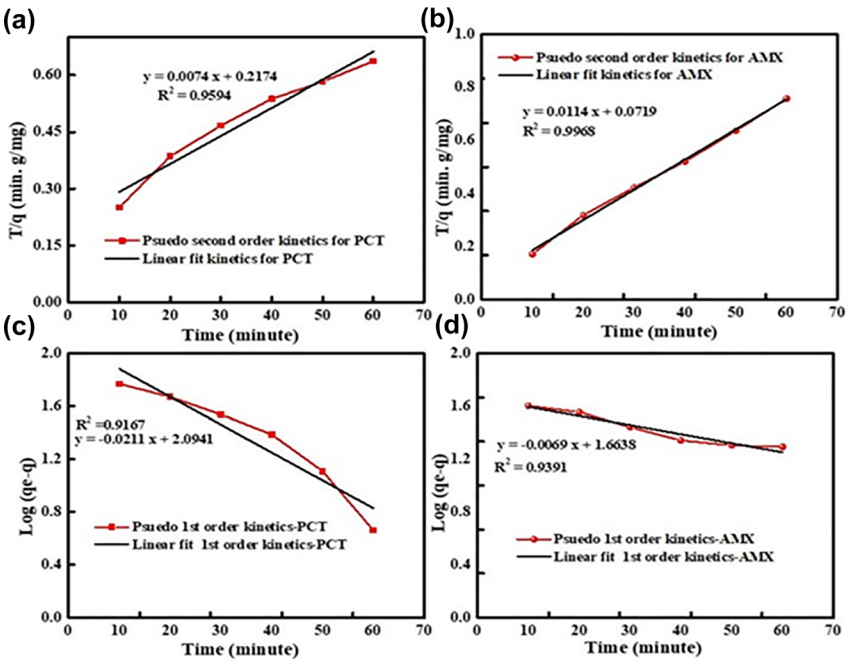

Figure 7: Pseudo second-order kinetics of (a, b) amoxicillin and paracetamol, and pseudo first order kinetics for (c, d) amoxicillin and paracetamol removal by PAC sorbent.

Table 2: Maximum adsorption capacity of paracetamol and amoxicillin of various adsorbent and present investigation.

Serial Number	Bio-sorbent	*q* (mg/g)	Reference
1	Beverage sludge activated carbon	57	[33]
2	Brazil nutshells derived activated carbon	409	[34]
3	Carbon derived from dehydrated sewage sludge	505	[35]
4	Dende-coconut and babassu coconut mesocarp	59	[36]
5	Sorbent derived from rice husk		[37]
6	Granular activated carbon from coconut	38.2	[38]
7	Yohimbe bark		[39]
8	Commercial activated carbon	221	[40]
9	Orange peels	10	[41]
10	Ground nutshell derived sorbent	3.0	[42]
11	Microalgae	53	[43]
12	Banana peels derived carbon	45	[44]
13	Activated carbon derived from tea waste	49	[45]
14	Grapes stalk derived sorbent		[46]
15	Sugarcane derived carbon	0.32	[47]
16	Peach seeds derived activated carbon	51.6	This work

4 Conclusions

Phosphoric acid treated activated carbon (PAC) was used as an adsorbent for the removal of amoxicillin and paracetamol and process variables (temperature, pH and reaction time) were optimized. At optimum conditions of process variables, the removal of 51.8 mg/g and 51.1 mg/g for the AMX and PCT, respectively was achieved. The sorption process followed the pseudo-second-order kinetics and Langmuir isotherm model and the AMX and PCT adsorption process was endothermic in nature. The activated charcoal materials can be a promising candidate to overcome the environmental problem due to AMX and PCT and PCA is also extendable for other pharmaceutical agents.

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