



Adsorption of Procion Red MX-5B dye from aqueous solution using homemade peach and commercial activated carbons

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Abstract

Wastewater treatment is of great concern in the textile industries because dyes, which are released into wastewater, are stable due to their structures and that some of the existing conventional techniques are not effective for aqueous removal of dyes. Among the advanced techniques for treatment of industrial effluents, adsorption is one of the most effective processes for removal of dyes from industrial wastewater. Similarly, activated carbons from different sources have been successfully applied as adsorbents for dye removal from aqueous solutions. In this context, this research evaluated the performance of activated carbon produced from peach pit (APC-1.0) for removal of a textile dye, Procion Red MX-5B (PR-5B), through batch adsorption process. The characteristic features of the peach activated carbon were obtained through Fourier transforms infrared spectroscopy, and scanning electron microscopy. The effectiveness of the activated to remove PR-5B from aqua system was compared with that of commercial activated carbon (CAC). In the kinetic study, the contact time was evaluated between 0 and 480 min using two different initial concentrations. Adsorption isotherms of PR-5B onto the two adsorbents were investigated in six different temperatures (298–326 K). The thermodynamic parameters of the process indicated spontaneous and exothermic adsorption. Equilibrium data were best described by Liu isotherm model with maximum removal capacities of 297.2 and 174.0 mg g⁻¹ for APC-1.0 and CAC, respectively.

Keywords Adsorption · Peach seed · Activated carbon · Isotherm · Microwave regeneration

Introduction

Effluents containing dyes, when released into rivers, can cause serious environmental problems. The presence of dyes in water is undesirable due to the lasting color and can cause a decrease in water transparency even at low concentrations, thereby preventing the penetration of sunlight and hindering the process of photosynthesis and increasing the chemical oxygen demand (Bae and Freeman 2007; Rosales et al. 2016). Furthermore, some dyes can cause allergies, cancer, and mutations (Alves de Lima et al. 2007; Demarchi et al. 2013).

The treatment of effluents containing dyes is one of the biggest challenges for the textile industry due to the fact that these substances have high chemical and structural stability, and are commonly persistent to the conventional wastewater treatments. Thus, it is usually necessary to apply more than one technology since each procedure has a limitation and the dyes are resistant to degradation and are photostable (Guarati and Zanoni 2000). Among the existing methods for

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the treatment of textile effluents, the adsorption has several advantages such as high color removal efficiency, low cost, ease of operation, continuous processing of large volumes of waste, and high possibility of regenerating spent adsorbent (Carneiro et al. 2010; Sharma et al. 2013; Rovani et al. 2014).

Due to the high stability of dyes, adsorption is one of the most efficient treatment process due to the easiness of design, operational simplicity and good efficiency in the removal of several type of pollutants, including dyes (Ribas et al. 2014; Rosales et al. 2016; Bazzo et al. 2016). In recent years, activated carbons have been mentioned by the Environmental Protection Agency of the USA as the best available control technology for wastewater treatment (de Franco et al. 2017). Although, there is a growing interest in search for alternative low-cost adsorbents, especially those prepared from wastes (SenthilKumar et al. 2010; Baccar et al. 2012; Dorado et al. 2014).

Brazil has a great agricultural potential and in this scenario the production of a large volume of waste can represent an environmental issue. The use of agricultural wastes to produce adsorbents is advantageous and economic alternative, which can be further used for water and wastewater treatment (Ribas et al. 2014).

In the present study, homemade peach activated carbon (APC), a low-cost adsorbent, was prepared and applied for the removal of Procion red, a textile dye, through batch adsorption process. Batch experiments were performed to investigate kinetic, equilibrium and thermodynamic aspects of the adsorption process. The adsorption potential of APC was compared with that of commercial activated carbon (CAC).

Materials and methods

Preparation of the adsorbent

Peach pit was collected, dried in an oven at 105 °C for 24 h, ground in a knife mill and properly stored for later use. For comparative purposes, all adsorption experiments were also carried out using commercial activated carbon (CAC) supplied by Merck (325–400 mesh) under the same experimental conditions of the synthesized peach carbon.

Carbon synthesis

The activated carbon was prepared using the following procedure: 70 g of organic component (peach pit powder), 70 g of inorganic components (28.6% clay, 28.6% lime, 14.3% of KOH, 14.3% Al (NO₃)₃ and 14.2% Na₂SO₄), and 50.0 mL of water were thoroughly mixed to obtain a homogeneous paste (Ribas et al. 2014; Saucier et al. 2015).

The resulting wet paste was placed in a cylinder of 4.8 by 14.0 cm and dried at room temperature for 24 h. The cylinder together with the paste was then placed in a stainless-steel reactor. The reactor was heated in the tubular furnace at 293 K min⁻¹ to 1073 K and remained at that temperature for 30 min. The resulting adsorbent was cooled to ambient temperature under Argon flow (25 mL min⁻¹), ground, sieved to a particle size ≤ 150 μm, and stored in a sealed container until used.

To remove inorganic compounds and improve the adsorption capacity of the produced activated peach carbon, chemical activation was performed on the adsorbent. The procedure used is summarized as: 10 g of the carbon was placed in contact with 200 mL of 6 mol L⁻¹ HCl in a 500-mL boiling flask, and the mixture was stirred on a magnetic stirrer for 2 h under reflux at 70 °C. The slurry was cooled down and filtered under vacuum using 0.45 μm membranes in a polycarbonate Sartorius system. The residue, after extensive washing with water (pH 2.0), was oven-dried at 100 °C for 5 h and stored until use. The adsorbent was named activated peach carbon (APC-1.0). The APC-1.0 contain peach pit (precursor) and inorganic components in a ratio 1:1. The ratio of the precursor to inorganics was varied during preliminary experiments, however, the composition of 1:1 (precursor/inorganic) gave the highest adsorption capacity, hence was used for further studies.

Characterization activated carbon

The activated carbon from peach was characterized using Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscope (SEM). The surface morphology of APC-1.0 was obtained using SEM (Hitachi, Tabletop microscope model TM3000, Tokyo, Japan). The functional groups present in APC-1.0 were visualized FTIR (Shimadzu Spectrometer, IR Prestige 21, Kyoto, Japan). For FTIR analysis, APC-1.0 and KBr were dried in an oven at 120 °C for 8 h and kept in a desiccator. The spectrum was obtained with a resolution of 4 cm⁻¹ with 100 cumulative scans.

Preparation of the solutions

The stock solution (5000 mg L⁻¹) of Procion Red MX-5B dye (CAS: 17804-49-8, C₁₉H₁₀Cl₂N₆O₇S₂, 615.33 g mol⁻¹, Sigma-Aldrich, purity 40%, Fig. 1) was prepared by dissolving 5.0 g of the dye in deionized water. All working solutions used in the experiments were obtained by dilution of the stock solution. The pH adjustment, when needed, was performed with addition of 0.1 mol L⁻¹ HCl or NaOH solution.

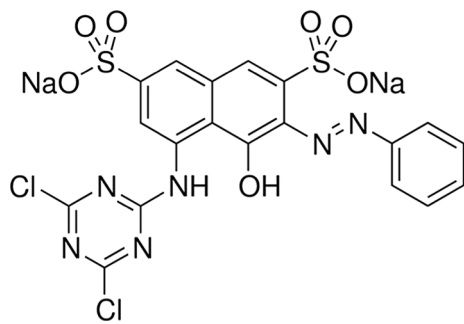


Fig. 1 Molecular structure of Procion Red MX-5B

Batch studies

Batch adsorption experiments were carried out in 50 ml Falcon tubes with 20 mL of dye solution and 50 mg of adsorbent. Samples were agitated in a thermostatic shaker at 150 rpm at pre-determined times. The mixtures were then centrifuged for 10 min at 15,000 rpm. To determine the residual dye in the supernatant, the solution was analyzed by UV–VIS spectrophotometer (T90, PG Instruments, London, United Kingdom) at a wavelength of 536 nm. When necessary, aliquots were diluted with an aqueous solution (water of suitable pH) before spectroscopic measurement. The amount of adsorbed dye and the percentage removal are calculated using Eqs. 1 and 2, respectively.

$$q = \frac{(C_0 - C_f)}{m} \cdot V \quad (1)$$

$$\% \text{Removal} = \frac{(C_0 - C_f)}{C_0} \cdot 100 \quad (2)$$

where q is the amount of dye adsorbed by the adsorbent in mg g^{-1} , C_0 is the initial dye concentration in contact with the adsorbent in mg L^{-1} , C_f is the final dye concentration after the adsorption process in mg L^{-1} , m is the mass of adsorbent in g and V is the volume of dye solution in L.

Kinect study

For the kinetic adsorption study, the contact times were varied between 0 and 480 min using two different initial concentrations (400 and 600 mg L^{-1}). These initial concentrations were selected because it is believed that in these conditions the saturation of the adsorbent material would be achieved.

The kinetic models used for evaluation and explanation of kinetic data were pseudo-first-order,

pseudo-second-order, general-order and intra-particle diffusion models. The respective models are represented by Eqs. 3–6.

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (3)$$

$$\frac{dq_t}{dt} = k_2(q_2 - q_t)^2 \quad (4)$$

$$q_t = q_e - \frac{q_e}{\left[k_N(q_e)^{n-1} \cdot t \cdot (n-1) + 1 \right]^{1/1-n}} \quad (5)$$

$$q_t = k_{id} \cdot t^{1/2} \quad (6)$$

where k_1 is the pseudo-first-order rate constant in min^{-1} ; k_2 is the pseudo-second-order rate constant in $\text{g mg}^{-1} \text{min}^{-1}$; k_n is the n th order rate constant in $\text{min}^{-1} (\text{g mg}^{-1})^{n-1}$; k_{id} is the intra-particle rate constant in $\text{mg g}^{-1} \text{min}^{-0.5}$, n is the order of reaction; q_e is the amount of dye adsorbed by the adsorbent at equilibrium in mg g^{-1} ; and q_t is the amount of dye adsorbed by the adsorbent at time, t , in mg g^{-1} .

Equilibrium study

Experiments were performed using different dye concentrations in the range 500–1700 mg L^{-1} to cover a wide range of concentrations since industrial waste disposal show varying concentrations. Studies were performed at six different temperatures, which ranged between 25 and 50 °C.

The mathematical models used to describe the adsorption isotherms were Langmuir (1918), Freundlich (1906) and Liu et al. (2003) as shown in Eqs. 7–9 respectively.

$$q = \frac{K \cdot Q_{\max} \cdot C_f}{1 + K \cdot C_f} \quad (7)$$

$$q_e = K_F \cdot C_f^{1/n} \quad (8)$$

$$q_e = \frac{Q_{\max} \cdot (K_g \cdot C_f)^{n_L}}{1 + (K_g \cdot C_f)^{n_L}} \quad (9)$$

Kinetic and equilibrium data were subjected to nonlinear fitting methods with successive interactions calculated by the Levenberg–Marquardt method. Interactions were also evaluated with the aids of the simplex method, based on the nonlinear fitting facilities of the Microcal Origin 9.0 software. A determination coefficient (R^2), an adjusted determination coefficient (R^2_{adj}) and an error function (F_{error}) were jointly used to evaluate the suitability of the models used

(Cardoso et al. 2011; Saucier et al. 2015). A F_{error} is defined as a measure of the differences between the theoretical and experimental amounts of dye adsorbed. The R^2 , R^2_{adj} and F_{error} are given by Eqs. 10–12, respectively.

$$R^2 = \left(\frac{\sum_i^n (q_{i,\text{exp}} - \bar{q}_{i,\text{exp}})^2 - \sum_i^n (q_{i,\text{exp}} - q_{i,\text{model}})^2}{\sum_i^n (q_{i,\text{exp}} - \bar{q}_{i,\text{exp}})^2} \right) \quad (10)$$

$$R^2_{\text{adj}} = 1 - (1 - R^2) \cdot \left(\frac{n-1}{n-p-1} \right) \quad (11)$$

$$F_{\text{error}} = \sqrt{\left(\frac{1}{n-p} \right) \cdot \sum_i^n (q_{i,\text{exp}} - q_{i,\text{model}})^2} \quad (12)$$

In these equations, $q_{i,\text{model}}$ represents individual theoretical q value predicted by the model; $q_{i,\text{exp}}$ represents individual experimental q value; \bar{q}_{exp} is the average of experimental q ; n represents the number of experiments while p represents the number of parameters in the fitting model (Cardoso et al. 2011; dos Reis et al. 2017).

Thermodynamic study

The thermodynamic parameters related to the adsorption process, standard Gibbs energy (ΔG° , kJ mol⁻¹), standard enthalpy change (ΔH° , kJ mol⁻¹) and entropy change (ΔS° , J mol⁻¹ K⁻¹), were determined using Eqs. 13–15. Equation 15 is obtained from Eqs. (13) and (14)

$$\Delta G^\circ = \Delta H^\circ - T \cdot \Delta S^\circ \quad (13)$$

$$\Delta G^\circ = -R \cdot T \cdot \ln(K) \quad (14)$$

$$\ln(K) = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{R} \frac{1}{T} \quad (15)$$

where R is the universal gas constant (8.314 J K⁻¹ mol⁻¹); T is the absolute temperature (K); K is the adsorption equilibrium constant at the isotherm that best described the equilibrium data. Different adsorption equilibrium constants can be obtained from different isotherm models (Calvete et al. 2010; Alencar et al. 2012; Cardoso et al. 2012; Dotto et al. 2012; Suksabye and Thiravetyan 2012). Thermodynamic parameters can also be calculated from the Liu equilibrium constant, K_g , as already reported (Prola et al. 2013; Adebayo et al. 2014; Ribas et al. 2014).

Microwave regeneration

The spent adsorbent was regenerated using microwave system. The regeneration system was made up of quartz

reactor loaded with pre-dried carbon, through which the gas flows. The temperature was controlled by two thermocouples. The microwave oven was operated at a frequency of 2.45 GHz with variable power from 0 to 1000 W provided by a microwave generator (Sairem). Microwaves were transmitted from the generator in a waveguide made of brass (Type 340, internal dimensions of 86.943 mm). This waveguide has a refrigerated cylinder with water inside, and it was used to absorb any reflected power. Temperature, heating rates, microwave power and gas flow rate were all controlled through a computer system using software developed just for this purpose (Fidalgo et al. 2014).

The heating ramp was 5 K min⁻¹ and N₂ gas was used with a flow rate of 16 cm³ min⁻¹. After reaching the desired temperature, the sample remained under heating for 60 min, and then the sample was cooled to room temperature. The carbon was regenerated.

Simulation of dye-house effluents

Two simulated dye-house effluents similar to industrial textile effluents at pH 2.0 were used to evaluate the adsorbent removal capacity. The synthetic dye-house effluent contained five typical dyes that are commonly used as fabric colorants and auxiliary chemicals. It is known that between 10 and 60% of synthetic dyes and almost all auxiliary chemicals remain in the dye bath (Hessel et al. 2007). The representative dyes, auxiliary chemicals and their concentrations are shown in Table 1.

Table 1 Chemical composition of the simulated dye-house effluents

| | Concentration (mg L ⁻¹) | |
|---------------------------------|-------------------------------------|------------|
| | Effluent A | Effluent B |
| Dyes | | |
| Procion Red MX-5B | 100 | 200 |
| Reactive Violet 5 | 20 | 40 |
| Evans Blue | 20 | 40 |
| Cibracon Brilliant Yellow 3G-P | 20 | 40 |
| Reactive Red 120 | 20 | 40 |
| Auxiliary chemicals | | |
| Na ₂ SO ₄ | 150 | 300 |
| NaCl | 150 | 300 |
| Na ₂ CO ₃ | 100 | 200 |
| CH ₃ COONa | 100 | 200 |
| CH ₃ OOH | 300 | 300 |
| Sodium dodecyl sulfate (SDS) | 106 | 106 |

Results and discussion

Characteristics of the activated peach carbon

The functional groups on the APC surface was obtained using FTIR spectroscopy. As shown in Fig. 2a, the major absorption bands present and their assignments are: 3380 cm^{-1} (O–H stretching vibration of alcohol), 1628 cm^{-1} (aromatic rings), 1103 cm^{-1} (C–O of alcohols and Si–O of silicates), and 789 cm^{-1} (C–H of aromatic out-of-plane bending). Some of these functional groups are responsible for adsorption of PR-5B dye from aqueous solution.

The SEM micrograph that shows the surface morphology of the APC-1.0 is presented in Fig. 2b. The particles of the adsorbent are aggregated. The porous and rough surface (characterized with irregular pores with different cavities) of the adsorbent is visible (Ribas et al. 2014; dos Reis et al. 2016). Chemical activation and leaching of inorganics from activated adsorbent have been found to increase the roughness, surface area and adsorption capacity of adsorbent (Ribas et al. 2014; Saucier et al. 2015; dos Reis et al. 2017).

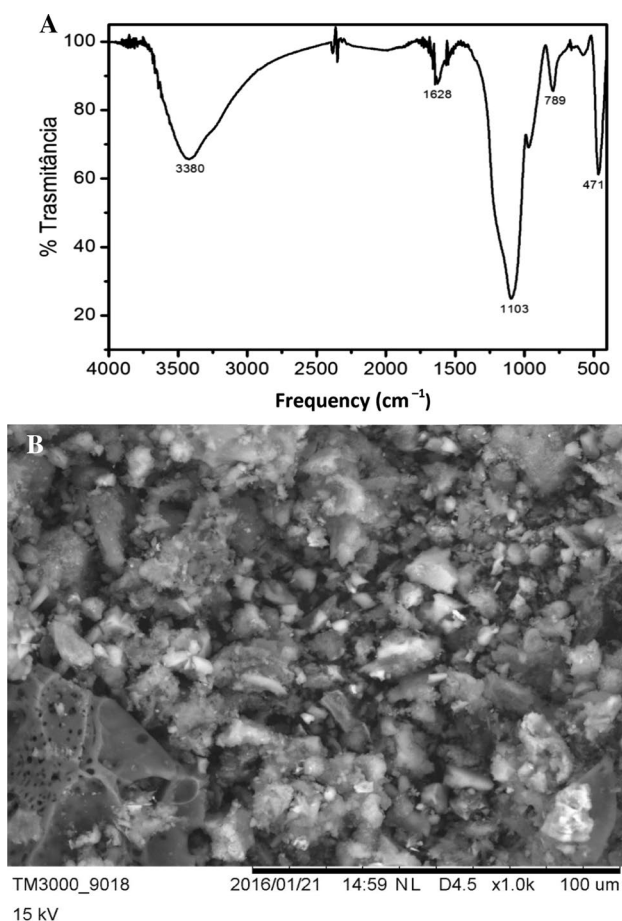


Fig. 2 The FTIR spectrum (a) and SEM micrograph (b) of APC-1.0

Effect of pH

Dyes usually have one pH range in which adsorption onto a certain material is favorable. The optimum pH value depends on the characteristics of the adsorbent and the interaction between the adsorbate and the solid surface. The pH effect was evaluated between 2 and 10 using 500 mg L^{-1} of Procion Red solution. The results obtained for both adsorbents, APC-1.0 and CAC, are shown in Fig. 3.

As can be seen in Fig. 3, the two adsorbents exhibit similar adsorption behavior. The APC-1.0 showed maximum percentage removal of 84.6% at pH 2. With the increase of pH, the percentage removal decreased until it reached its minimum value of 64.4% at pH 10. For the commercial activated carbon (CAC), no significant variation in the percentage removal was observed between pH 2 and 5, with an average percentage removal of 75%. However, the adsorption removal decreased with the increase of pH until it reached a minimum value of 61.2% at pH 10. Adsorption capacity of cationic dyes tends to increase at relatively high pH values whereas for the anionic dyes, the adsorption process is favored at low pH (Hubbe et al. 2012). The initial pH of all dye working solutions was set at 2 for subsequent experiments.

Kinetic studies

The kinetic experimental data were analyzed using pseudo-first-order, pseudo-second-order, general-order, and intra-particle diffusion kinetic models. The nonlinear curves for both adsorbents are presented in Fig. 4. Table 2 shows the parameters obtained for the kinetic models using APC-1.0 and CAC in two different initial concentrations. As can be

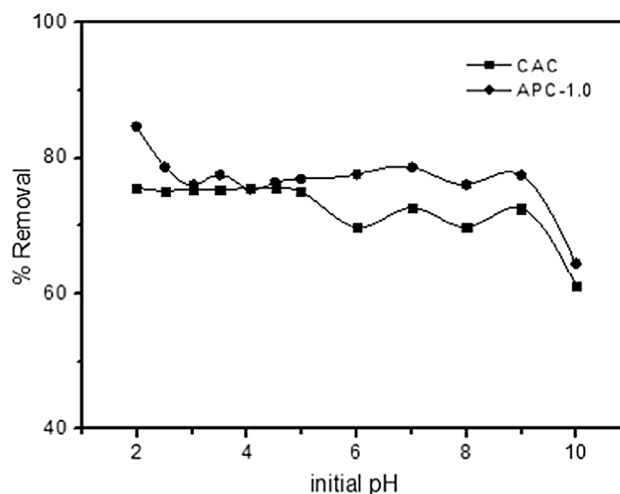


Fig. 3 Dependence of pH on the adsorption capacity of PR-5B dye on CAC and APC-1.0. Conditions: temperature, 298 K; adsorbent mass, 50.0 mg; time, 8 h; pH, 2–10; dye concentration, 500 mg L^{-1}

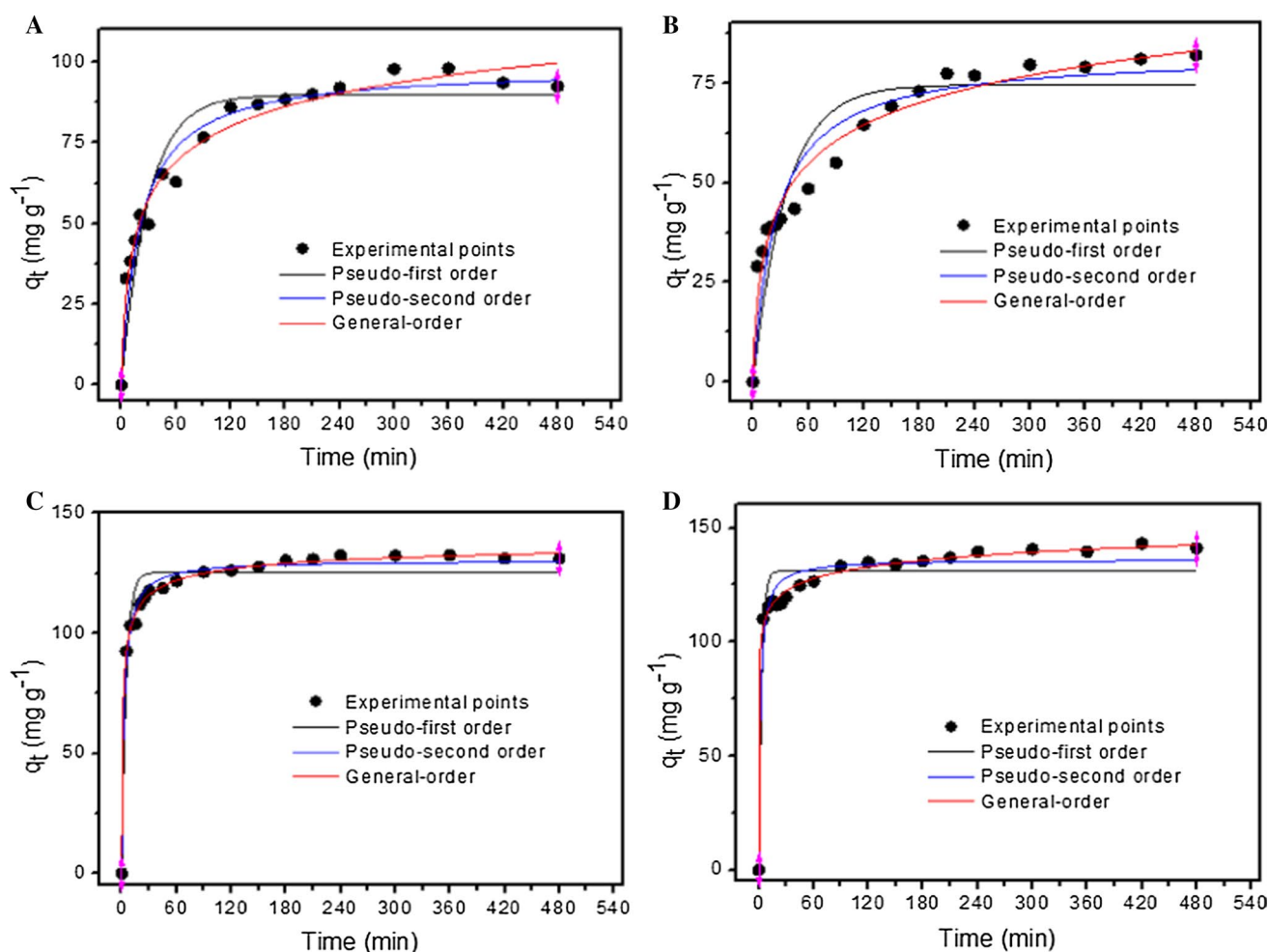


Fig. 4 Kinetic isotherm curves of a CAC and PR-5B dye at 400 mg L⁻¹; b CAC and PR-5B dye at 600 mg L⁻¹; c APC-1.0 and PR-5B at 400 mg L⁻¹; and d APC-1.0 and PR-5B at 600 mg L⁻¹. Conditions:

initial pH, 2; temperature, 25 °C; adsorbent mass, 50 mg; and contact times, 0–480 min

observed in Fig. 4 and Table 2, experimental data were best fitted by the general-order kinetic model. Compared to other kinetic models, the general-order model showed the highest values of R_{adj}^2 for both APC-1.0 and CAC.

To choose a particular model that best-described adsorption process, R_{adj}^2 values alone are not enough for judgement. It is therefore important to consider standard deviation (F_{error}) because this parameter evaluates the difference between each data point measured experimentally and theoretical data point predicted by model (Alencar et al. 2012; Adebayo et al. 2014). Hence, F_{error} values were analyzed. For the pseudo-first-order model F_{error} ranged from 5.99 to 8.25 for APC-1.0 and from 6.22 and 6.52 for CAC. For the pseudo-second-order model, F_{error} ranged from 2.39 to 5.16 and 4.16 to 3.55 for APC-1.0 and CAC, respectively. For the general-order kinetic model, F_{error} ranged between 1.14 and 1.83 for APC-1.0 and from 3.55 to 4.16 for CAC. The observation indicates that the general-order model best

explained the adsorption process of the PR-5B dye onto both activated carbons.

It is pertinent to discuss some of the parameters of general-order model, which is the best kinetic model in this study. The values of rate constant, k_n , decreased for the two adsorbents when the concentration of the dye was increased. The reaction was faster when few molecules of the dye was present in the solution, as the number of molecules (concentration) increased to compete for relatively the same number of active sites, the reaction rate decreased. However, the initial rate constant, h_o , increased when the concentration was increased. In another way, when the initial concentration was increased by 1.5-fold, the half-lives (the times require to attain 50% of q_e) of adsorption process increased by ca. 3.9-fold and 1.4-fold for APC-1.0 and CAC, respectively. That is, the time to reach 50% of equilibrium amount increased with concentration. This observation is expected because there

Table 2 Kinetic parameters PR-5B dye adsorption onto APC-1.0 and CAC

| | APC-1.0 | | CAC | |
|---|------------------------|------------------------|------------------------|------------------------|
| | 400 mg L ⁻¹ | 600 mg L ⁻¹ | 400 mg L ⁻¹ | 600 mg L ⁻¹ |
| Pseudo-first-order | | | | |
| k_1 (min ⁻¹) | 0.2009 | 0.3115 | 0.0345 | 0.0282 |
| q_e (mg g ⁻¹) | 125.3 | 131.1 | 89.9 | 74.6 |
| h_0 (min ⁻¹) | 25.2 | 40.8 | 3.1 | 2.1 |
| $t_{1/2}$ (min) | 3.4 | 2.2 | 20.0 | 24.6 |
| R^2_{adj} | 0.9329 | 0.9138 | 0.9034 | 0.8376 |
| F_{error} (mg g ⁻¹) | 5.99 | 8.25 | 6.22 | 6.52 |
| Pseudo-second-order | | | | |
| k_2 (g mg ⁻¹ min ⁻¹) | 2.87×10^{-3} | 3.92×10^{-3} | 4.99×10^{-4} | 4.83×10^{-4} |
| q_e (mg g ⁻¹) | 130.4 | 136.1 | 98.38 | 82.38 |
| h_0 (mg g ⁻¹ min ⁻¹) | 48.81 | 72.61 | 4.830 | 3.278 |
| $t_{1/2}$ (min) | 2.723 | 1.752 | 20.42 | 25.21 |
| R^2_{adj} | 0.9870 | 0.9651 | 0.9578 | 0.9172 |
| F_{error} (mg g ⁻¹) | 2.39 | 5.16 | 4.24 | 4.64 |
| General-order | | | | |
| k_n (min ⁻¹ (g mg ⁻¹) ⁿ⁻¹) | 1.49×10^{-6} | 8.83×10^{-31} | 1.74×10^{-19} | 1.24×10^{-29} |
| q_e (mg g ⁻¹) | 143.3 | 230.1 | 199.01 | 226.06 |
| n | 3.723 | 14.50 | 8.610 | 12.66 |
| h_0 (mg g ⁻¹ min ⁻¹) | 108.7 | 153.2 | 10.81 | 6.375 |
| $t_{1/2}$ (min) | 1.883 | 7.384 | 19.65 | 27.86 |
| R^2_{adj} | 0.9972 | 0.9957 | 0.9740 | 0.9657 |
| F_{error} (mg g ⁻¹) | 1.14 | 1.83 | 4.16 | 3.55 |
| Intra-particle diffusion | | | | |
| $k_{id,2}$ (mg g ⁻¹ min ^{-0.5})* | 4.219 | 2.908 | 2.473 | 4.164 |

Conditions: initial pH, 2; temperature, 25 °C; adsorbent mass, 50 mg; and contact times, 0–480 min
 *Second stage

are more molecules of the dye at 600 mg L⁻¹ than at 400 mg L⁻¹, hence more time is needed for the process to be half-completed at 600 mg L⁻¹ than at 400 mg L⁻¹.

The results from the kinetic study and the effect of pH indicated that the PR-5B adsorption mechanism onto APC-1.0 and CAC involves ion exchange mechanisms. To confirm the existence of electrostatic attractions, thermodynamic study was conducted.

To determinate the influence of mass transfer resistance in the PR-5B adsorption on the two adsorbents, the intra-particle diffusion model was used. The graph of q_t versus $t^{1/2}$ is shown in Fig. 5. The adsorption of PR-5B showed three steps, which are represented by three linear portions of the curve. The first linear portion is the fastest step and it is related to the transport of dye molecules to the adsorbent surface. The second portion is a slow process and it is attributed to intra-particle diffusion. The third and final step represents the stage when the diffusion through the smaller pores decreases and the equilibrium is reached (Calvete et al. 2010; de Franco et al. 2017). This kinetic profile in three well-defined linear portions reveals that adsorption is controlled by the resistance mass transfer.

According to the data presented, the minimum contact times for equilibrium to occur between the dye and the adsorbent are 120 min for APC-1.0 and 300 min for CAC. In the equilibrium experiments, the contact time was increased to ensure that equilibrium is reached even at high concentrations of the dye (Prola et al. 2013; Ribas et al. 2014).

Adsorption isotherms

Six temperatures in the range between 25 and 50 °C were studied and the equilibrium experimental data were fitted into Langmuir, Freundlich and Liu isotherms (Table 3). Isotherms at 25 °C for the PR-5B adsorption onto APC-1.0 and CAC are shown in Fig. 6. As seen in Table 3, for all temperatures evaluated, the adsorption equilibrium of PR-5B dye was best described by Liu model because the model exhibited lowest standard deviation indices among the three models. The F_{error} values of Liu model ranged from 2.75 to 6.55 for APC-1.0 and from 2.34 to 4.88 for CAC. The maximum adsorption capacities (Q_{max}) of PR-5B were obtained at 25 °C for both adsorbents tested—297.2 mg g⁻¹ for APC-1.0 and 174 mg g⁻¹ for CAC. This result revealed that the

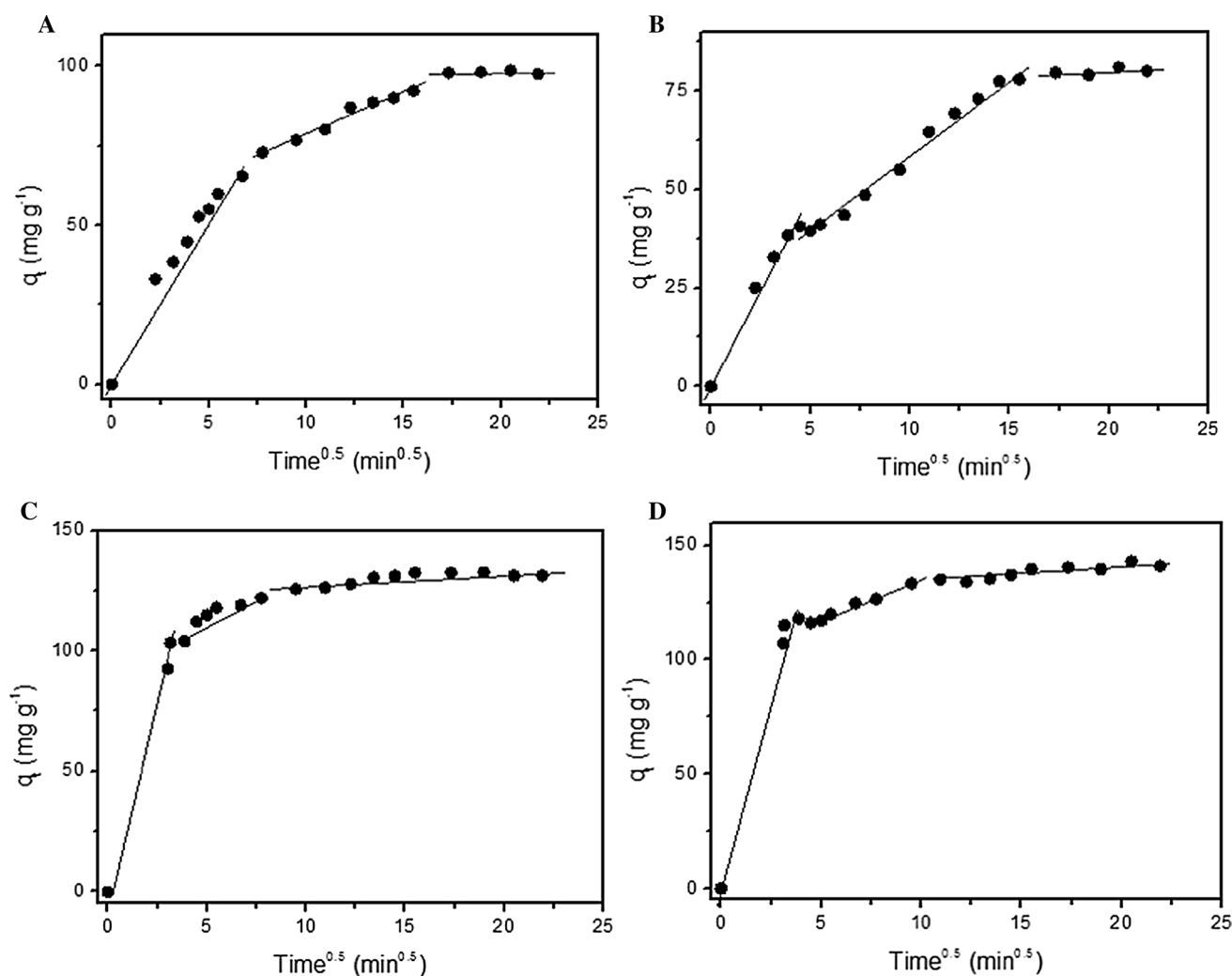


Fig. 5 Kinetic curves of intra-particle diffusion model: a 400 mg L⁻¹ of PR-5B and CAC as adsorbent; b 600 mg L⁻¹ of PR-5B and CAC as adsorbent; c 400 mg L⁻¹ of PR-5B and APC-1.0 as adsorbent; and d 600 mg L⁻¹ of PR-5B and APC-1.0 as adsorbent

activated peach carbon had higher adsorption capacity than the commercial activated carbon under the same experimental conditions.

The best model (Liu model) for description of equilibrium data in this study, combined Langmuir and Freundlich isotherm models, however, the infinite adsorption assumption from the Freundlich model and the monolayer assumption of Langmuir model were jettisoned (Liu et al. 2003). The adsorbate molecules may prefer and occupy certain active sites on the surface of adsorbent (Liu et al. 2003; Lima et al. 2015). As shown in Table 3, the Liu equilibrium constant, K_g (L mg⁻¹), decreases as temperature increases. This signifies that an increase in temperature does not favor the adsorption process, hence, an exothermic process. The values n_L (dimensionless exponent of the Liu model) assume positive values and range from 0.8516 to 3.262 for APC-1.0 from 0.9659 to 4.055 for CAC.

Thermodynamic study

The results of thermodynamic study obtained for PR-5B adsorption onto APC-1.0 and CAC are shown in Table 4. According to Table 4, the negative values of ΔG° reveal that the adsorption onto both adsorbents occurred spontaneously. The negative values of ΔH° indicate the process was exothermic and that the adsorption process exhibited physical characteristics (physisorption) (Sun and Wang 2010; Rathod et al. 2015). Negative values of ΔS° indicate that with an increase in temperature, there is a reduction in system instability at the solid/fluid interface, i.e., the dye molecules exhibit more ordered layers on the surface of the adsorbent (Gupta et al. 2011).

Table 3 Isotherm parameters for PR-5B dye adsorption using APC-1.0 and CAC

| | Temperature | | | | | |
|--|-------------|--------|--------|--------|--------|--------|
| | 298 K | 303 K | 308 K | 313 K | 318 K | 323 K |
| <i>APC-1.0</i> | | | | | | |
| Langmuir | | | | | | |
| Q_{max} (mg g ⁻¹) | 272.7 | 155.7 | 152.6 | 163.0 | 182.6 | 231.9 |
| K_L (L mg ⁻¹) | 0.0153 | 0.0239 | 0.0310 | 0.0254 | 0.0157 | 0.0110 |
| R^2_{adj} | 0.9922 | 0.9774 | 0.9809 | 0.9616 | 0.9781 | 0.9862 |
| F_{error} (mg g ⁻¹) | 8.23 | 5.64 | 4.97 | 7.37 | 4.49 | 5.67 |
| Freundlich | | | | | | |
| K_F (mg g ⁻¹ (mg L ⁻¹) ^{-1/nF}) | 84.69 | 83.63 | 86.46 | 77.20 | 71.34 | 48.03 |
| n_F | 6.054 | 11.72 | 12.57 | 9.528 | 7.774 | 4.485 |
| R^2_{adj} | 0.9697 | 0.9736 | 0.9796 | 0.9471 | 0.9507 | 0.9791 |
| F_{error} (mg g ⁻¹) | 4.33 | 5.75 | 4.79 | 7.90 | 5.74 | 5.31 |
| Liu | | | | | | |
| Q_{max} (mg g ⁻¹) | 297.2 | 188.1 | 179.29 | 172.7 | 166.3 | 243.9 |
| K_g (L mg ⁻¹) | 0.0258 | 0.0209 | 0.0175 | 0.0142 | 0.0117 | 0.0098 |
| n_L | 1.643 | 2.283 | 1.553 | 3.262 | 3.153 | 0.8516 |
| R^2_{adj} | 0.9972 | 0.9772 | 0.9794 | 0.9732 | 0.9945 | 0.9854 |
| F_{error} (mg g ⁻¹) | 2.75 | 5.59 | 4.34 | 6.55 | 3.21 | 5.11 |
| <i>CAC</i> | | | | | | |
| Langmuir | | | | | | |
| Q_{max} (mg g ⁻¹) | 201.8 | 163.1 | 248.7 | 194.1 | 221.3 | 234.7 |
| K_L (L mg ⁻¹) | 0.0146 | 0.0335 | 0.0089 | 0.0241 | 0.0193 | 0.0115 |
| R^2_{adj} | 0.9853 | 0.98 | 0.9881 | 0.9749 | 0.9923 | 0.9748 |
| F_{error} (mg g ⁻¹) | 4.21 | 4.14 | 5.98 | 6.35 | 3.80 | 6.68 |
| Freundlich | | | | | | |
| K_F (mg g ⁻¹ (mg L ⁻¹) ^{-1/nF}) | 73.35 | 109.5 | 45.80 | 88.54 | 87.65 | 55.44 |
| n_F | 7.197 | 18.82 | 4.208 | 9.038 | 7.652 | 4.929 |
| R^2_{adj} | 0.9701 | 0.9698 | 0.9812 | 0.9492 | 0.9788 | 0.9410 |
| F_{error} (mg g ⁻¹) | 5.77 | 4.71 | 6.66 | 8.17 | 5.67 | 9.33 |
| Liu | | | | | | |
| Q_{max} (mg g ⁻¹) | 174.0 | 156.3 | 153.4 | 142.2 | 106.9 | 146.8 |
| K_g (L mg ⁻¹) | 0.0184 | 0.0146 | 0.0119 | 0.0096 | 0.0072 | 0.0054 |
| n_L | 2.775 | 4.055 | 0.9659 | 2.234 | 1.916 | 2.174 |
| R^2_{adj} | 0.9946 | 0.9932 | 0.9871 | 0.9866 | 0.9964 | 0.9915 |
| F_{error} (mg g ⁻¹) | 3.24 | 2.34 | 5.62 | 4.73 | 3.15 | 4.88 |

Conditions: initial pH, 2; adsorbent mass, 50.0 mg; initial PR-5B concentrations 500–1700 mg L⁻¹

Microwave regeneration

Regeneration experiments were performed on APC-1.0 in a series of adsorption–regeneration cycles and their results are shown in Table 5. The adsorption efficiency of PR-5B gradually decreased at every regeneration cycle. In each cycle, there was a loss of ca. 12% in the adsorbent mass. Thus, a decrease in regeneration efficiency occurred due to incomplete dye removal from the solid phase and reactions on the surface of the adsorbent which can reduce the number and size of the pores or even damages on the carbon structure (Guo et al. 2011; Salame and Bandosz 1999).

However, in all the adsorption–regeneration cycles, the efficiencies were significant. After the first cycle, ≈84% of removal was obtained and after the 4th cycle the efficiency was still significant, about 40%. The results showed the microwave technique used for regeneration of the saturated adsorbent, with PR-5B, is a viable method. Microwave technique was successfully used to regenerate bamboo charcoal (Liao et al. 2013). Sun et al. (2017) also studied microwave regeneration of activated carbon and obtained high percentages of recoveries after 5 cycles with a weight loss close to 20%. The authors cited that the regeneration can be enhanced with the use of ultraviolet radiation.

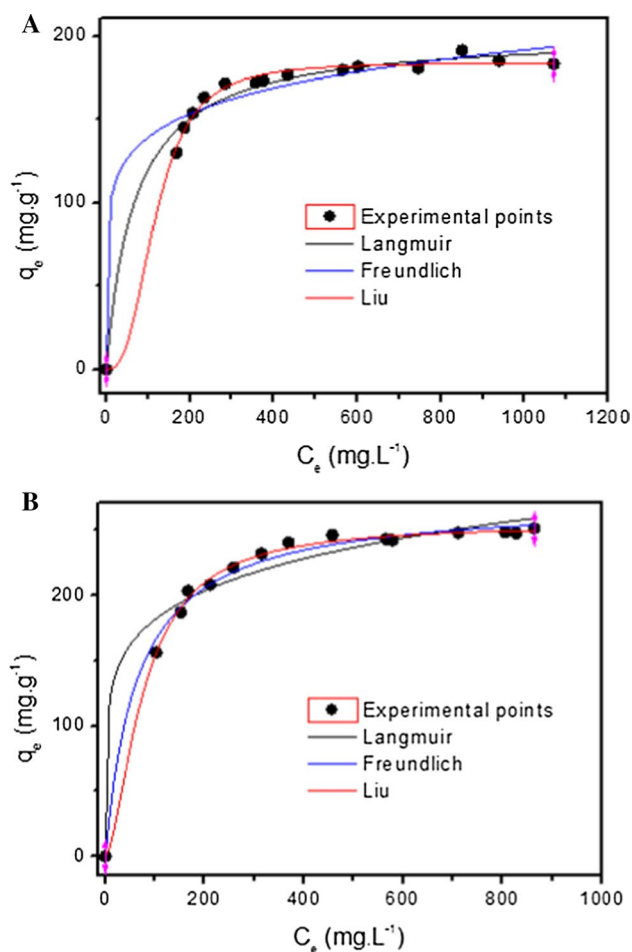


Fig. 6 Isotherm curves of adsorption of PR-5B onto a CAC and b APC-1.0 at 25 °C. Conditions: initial pH, 2; adsorbent mass, 50.0 mg; initial PR-5B concentrations 500–1700 mg L⁻¹

Table 4 Thermodynamic parameters of PR-5B dye adsorption onto APC-1.0 and CAC

| | Temperature | | | | | |
|---|----------------------|---------------------|----------------------|---------------------|---------------------|---------------------|
| | 298 K | 303 K | 308 K | 313 K | 318 K | 323 K |
| APC-1.0 | | | | | | |
| K_g (L mol ⁻¹) | 1.588×10^4 | 1.286×10^4 | 1.0768×10^4 | 8.738×10^3 | 7.199×10^3 | 6.03×10^3 |
| ΔG° (kJ mol ⁻¹) | -23.96 | -23.84 | -23.77 | -23.62 | -23.48 | -23.37 |
| ΔH° (kJ mol ⁻¹) | -31.07 | | | | | |
| ΔS° (J K ⁻¹ mol ⁻¹) | -23.82 | | | | | |
| R_{adj}^2 | 0.9995 | | | | | |
| CAC | | | | | | |
| K_g (L mol ⁻¹) | 1.1322×10^4 | 8.983×10^3 | 7.322×10^3 | 5.907×10^3 | 4.443×10^3 | 3.323×10^3 |
| ΔG° (kJ mol ⁻¹) | -23.13 | -22.93 | -22.79 | -22.60 | -22.20 | -21.77 |
| ΔH° (kJ mol ⁻¹) | -38.67 | | | | | |
| ΔS° (J K ⁻¹ mol ⁻¹) | -51.85 | | | | | |
| R_{adj}^2 | 0.9911 | | | | | |

Conditions: mass of adsorbent, 50 mg; pH, 2; temperature, 298–323 K

Table 5 Regeneration of the adsorbent for each adsorption–desorption cycle

| Cycle | q/q_0 (%) |
|-------|------------------|
| 1 | 83.94 ± 5.18 |
| 2 | 59.63 ± 2.77 |
| 3 | 47.21 ± 4.12 |
| 4 | 40.74 ± 3.87 |

Dye-house effluents

Adsorption efficiencies of activated carbon produced from peach pit (APC-1.0) and commercial activated carbon (CAC) for the removal of PR-5B were determined using synthetic effluent solutions shown in Table 1. The UV–Vis spectra of the solutions were analyzed at wavelengths between 300 and 750 nm. The absorbance bands used to quantitate the color removal efficiency of the effluents are shown in Fig. 7. The APC-1.0 adsorbent removed 94.7% of the compounds present in the effluent A and 88.4% of the effluent B while CAC removed 82.3% and 51.4% of the effluent A and B, respectively. Both adsorbents were efficient and achieved high removal percentages of the textile dye. However, for the treatment of effluent B (effluent with high concentrations of dyes and auxiliary chemicals), APC-1.0 performed significantly better than CAC. When compared with other adsorbents reported in literature, the APC-1.0 shows high effectiveness for industrial wastewater treatment (Adebayo et al. 2014; Cardoso et al. 2012; Prola et al. 2013).

Comparison of adsorption capacities of different adsorbents

It is difficult to compare the adsorption capacities of different adsorbents for removal of a dye because dye of the same chemical structure can have different commercial names

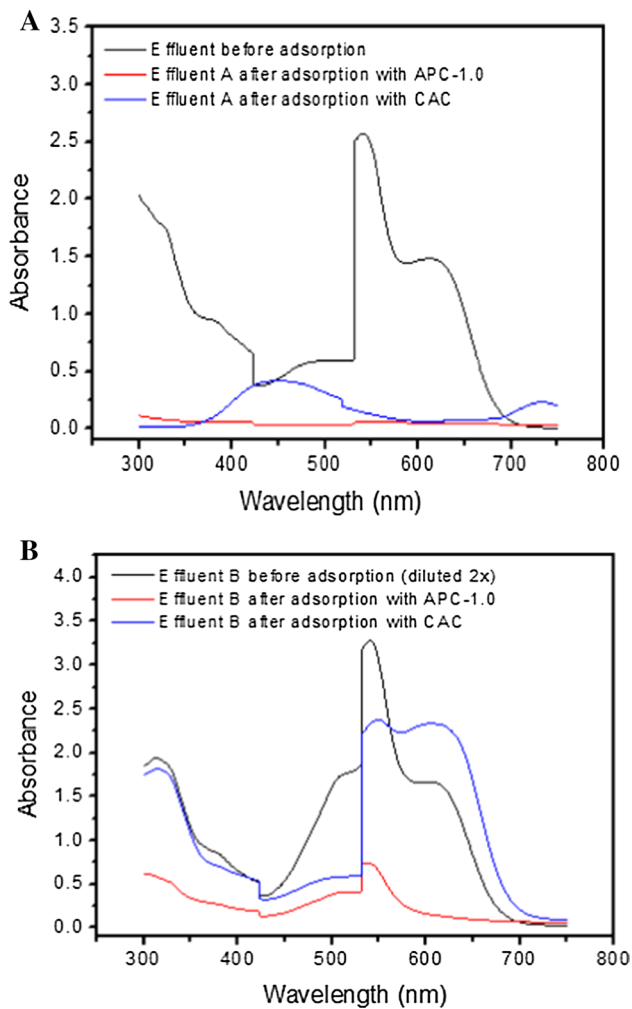


Fig. 7 UV/Visible spectra of simulated dye effluents before and after treatment with APC-1.0 and CAC. a Effluent A; and b effluent B

depending on the manufacturers. However, Table 6 presents maximum adsorption capacities of different adsorbents from various studies at optimized conditions. Regarding the differences in the experimental conditions of the cited references, the activated peach carbon showed excellent adsorption capacity. It is observed that APC-1.0 had higher Q_{max} than CAC. Thus, the activated carbon reported in this work can be successfully used for the treatment of dye effluents.

Conclusions

Activated peach carbon (APC-1.0) was prepared from peach pit and its adsorption efficiency was tested for removal of a textile dye, Procion red MX-5B. Batch experiments were performed to investigate the kinetic, equilibrium and thermodynamic aspects of the study, and the results were compared with those of commercial activated carbon (CAC) using the same experimental conditions. The contact times for adsorption of PR-5B dye to reach equilibrium were 120 and 300 min for APC-1.0 and CAC, respectively, at pH 2. The adsorption process was best described by the general-order kinetic model. The intra-particle diffusion model was used to investigate the stages of adsorption, and the kinetic data showed that adsorption onto the two adsorbents exhibited three stages. In the investigation of isotherms, Liu model was the best model for description of equilibrium data at six different temperatures ranging from 25 to 50 °C. The maximum adsorption uptake was obtained at 25 °C—297.2 mg g⁻¹ for APC-1.0 and 174 mg g⁻¹ for CAC. Thermodynamic adsorption parameters revealed that the adsorption process was exothermic ($\Delta H^\circ < 0$), and spontaneous ($\Delta G^\circ < 0$) for both adsorbents. The negative values of ΔS° indicated that an increase in temperature reduced the system disorder.

Table 6 Maximum adsorption capacities of different adsorbents used for removal of various dyes

| Adsorbent | Dye adsorbate | Q_{max} (mg g ⁻¹) | References |
|--|----------------------|---------------------------------|------------------------|
| Carboxy-methyl Lignin-Al | Reactive blue MX-R | 335.7 | Adebayo et al. (2014) |
| <i>Thamnidium elegans</i> | Reactive red 198 | 234.2 | Akar et al. (2013) |
| SAH | Acid yellow 36 | 200.0 | Patel and Patel (2013) |
| SAH | Acid red 73 | 198.1 | Patel and Patel (2013) |
| Peanut husk treated with HCl | Drimarine black CL-B | 51.0 | Noreen et al. (2013) |
| Commercial activated carbon | Reactive red 120 | 267.2 | Cardoso et al. (2012) |
| Cupuassu shell | Reactive red 194 | 64.1 | Cardoso et al. (2011) |
| Cupuassu shell | Direct blue 53 | 37.5 | Cardoso et al. (2011) |
| Papaya stem | Methylene blue | 261.0 | Krishni et al. (2014b) |
| Banana leaves | Methylene blue | 109.9 | Krishni et al. (2014a) |
| Commercial activated carbon | Reactive violet 5 | 517.1 | Ribas et al. (2014) |
| Acidified cocoa shell activated carbon | Reactive violet 5 | 603.3 | Ribas et al. (2014) |
| Commercial activated carbon (CAC) | Procion red MX-5B | 174.0 | This work |
| Activated peach carbon (APC-1.0) | Procion red MX-5B | 297.2 | This work |

Microwave regeneration tests with APC-1.0 were successfully conducted. After four adsorption–regeneration cycles, the adsorbent exhibited satisfactory adsorption capacities. The APC-1.0 effectively removed over 88% of textile dyes from synthetic dye-house effluents while CAC removed ca. 82%.

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Compliance with ethical standards

Conflict of interest All authors declare no conflict of interest in the publication of this paper.

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