

Acid Blue 25 Adsorption onto Phosphoric Acid Treated Rubber Leaf Powder

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Abstract The performance of rubber leaf powder treated with phosphoric acid in the removal of Acid Blue 25 (AB25) from aqueous solution was evaluated. The functional groups on the adsorbent surface were confirmed by Fourier transform infrared (FT-IR) spectroscopy. The effects of several important parameters that can affect adsorption capacities such as pH, agitation period, adsorbent dosage and different initial concentrations of AB25 were studied. In order to describe adsorption isotherm of phosphoric acid treated rubber leaf powder (PARL), the sorption data were analyzed using a linear form of Langmuir and Freundlich equation. The maximum adsorption capacity determined from Langmuir isotherm was 28.09 mg/g at 320 K. Two kinetic models including pseudo-first order and pseudo-second order were used to analyse the AB25 adsorption process, and the result showed that the pseudo-second order fitted well with correlation coefficients greater than 0.99. Therefore, results showed that PARL are favourable adsorbent to be used to remove the AB25 dye.

Keywords Acid Blue 25, Adsorption, Phosphoric acid, Rubber leaf powder, FT-IR isotherm, Kinetic

1. Introduction

The industries are reported to have been releasing about 2 % of dyes into the ecosystem through their effluents of which they are non-biodegradable and being accumulated in the natural water bodies [1]. Textile, paper mills and leather are among the major industries that cause serious environmental problems [2]. The colouration of the water by the presence of dyes even in small concentration is easily detectable and may have an inhibitory effect on the process of photosynthesis [3], thus can affect the aquatic ecosystem. Removing synthetic dyes from the industrial effluent is of great challenge since most dyes are stable to light and heat, high in organic content and have complex aromatic structures. Therefore, various conventional methods have been carried out to remove these soluble coloured contaminants from aqueous solutions. Among all methods, adsorption is known to be the most efficient process to sequester organic compounds from aqueous streams [4], and activated carbon is the most commonly used adsorbent but it is quite expensive [5]. A number of low cost adsorbents have been employed for removal studies of dyes and chemical activation by using phosphoric acid had been attempted to improve pores structure.

Rubber trees are widely planted in Malaysia and have become one of the major plantation crops after oil palm. Rubber related industries are now expanding to meet the demands from consumers. However, the vast area of rubber plantation in Malaysia generates an enormous amount of 'waste' rubber leaves especially during the dry season (February to March) every year [6]. The application of rubber leaves as an adsorbent for the removal of various heavy metal ions from wastewater have been studied and reported in the literature [6-9]. The physical modification of the rubber leaf powder can provide low surface area, an indicative of low porosity of adsorbent. The average pores diameter suggesting that the treated rubber leaf powder is a mesoporous adsorbent [7]. Despite rubber leaf powder good adsorption capability, further chemical modification could be done to enhance its ability. However no studies have been conducted on phosphoric acid treated rubber leaf powder.

In this study, the adsorption of Acid Blue 25 (AB25) onto phosphoric acid treated rubber leaf powder (PARL) was investigated. Experiments were carried out as a function of pH, agitation period, the effect of adsorbent dosage and temperature on adsorption efficiency of AB25. The equilibrium adsorption data of AB25 onto PARL powder were determined using Langmuir and Freundlich equations. Pseudo-first order and pseudo-second order kinetic models were used to determine the adsorption rates.

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2. Materials and Method

2.1. Materials

Dried mature (brownish colour) rubber leaves were obtained from Universiti Teknologi MARA Pahang, Malaysia rubber plantation. All the reagents used throughout the experiments were analytical-reagent grade and utilized without any further purification. Deionised distilled water was used to prepare all solutions.

2.2. Preparation of Phosphoric Acid Treated Rubber Leaf Powder

Rubber leaves were ground with a mechanical grinder and sieved to obtain a particle size of $< 500 \mu\text{m}$. Then, 75 mL of 0.5 M H_3PO_4 solution was added into the flask containing 5 g of the dried rubber leaf powder. The mixture was stirred using a magnetic stirrer at 300 rpm for 1 h, and later repeatedly washed with distilled water. The rubber leaf powder was then filtered using filter paper by gravity filtration. The filtrate was kept for pH testing to ensure that it remained unchanged. The adsorbent was dried in an oven at 80°C overnight, sieved to obtain particle size approximately $< 300 \mu\text{m}$ before used. The powder was designated as PARL. The structure of PARL before and after AB25 adsorption obtained was characterized by a FT-IR (Perkin-Elmer System 2000 Model, USA) spectrometer.

2.3. Batch Adsorption Experiments

A stock solution of 500 mg/L of AB25 dye solution was freshly prepared by weighing 0.25 g and dissolved in 500 mL volumetric flask and finally top up with deionised distilled water until the mark. The absorbance of the standard AB25 solution was determined using an ultraviolet-visible spectrophotometer (Perkin Elmer Lambda 35, USA) at a maximum wavelength of 601 nm.

The effect of initial pH of the solution on the adsorption of AB25 onto PARL was performed in the pH range of 2-10. Five sets of 100 mL AB25 dye solutions with a concentration of 20 mg/L were prepared. NaOH and HCl solution with appropriate concentrations were used to adjust the pH to ensure that there was no change in the concentration of the solution. Then, 0.10 g of PARL was transferred into the solution and left for shaking by using Heidolph Unimax 1010 shaker for 2 h at the particular pH. The pH that gave the highest adsorption capacity was used for the rest of the study. The effect of agitation time was also studied to determine the optimum condition for the adsorption of AB25 by varying the agitation time from 30-180 min at pH 2. The effect of adsorbent dosage was investigated by varying the initial mass of the adsorbent in the range of 0.01-0.1 g. The percentage removal of AB25 was calculated by the following equation:

$$\text{Removal (\%)} = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

where C_o is the initial concentration and C_e is the final concentration of AB25 (mg/L) respectively.

Adsorption equilibrium studies were conducted at optimum condition where the pH is 2 and the agitation time is 120 min. for PARL at 300 K. Isotherm studies were conducted with a constant amount of PARL and varying the initial concentration of AB25 in the range of 20-50 mg/L.

The amount of adsorption capacity at equilibrium was calculated using the following equation:

$$\text{Adsorption capacity, } q_e = \frac{C_o - C_e}{m} v \quad (2)$$

where q_e is the adsorption capacity at equilibrium (mg/g), C_o is the initial concentration of AB25 (mg/L), C_e is the final concentration of AB25 (mg/L), v is the volume of AB25 (mL) and m is the weight of the PARL (g).

For batch kinetic studies, 0.10 g PARL were equilibrated at optimum condition used previously. The PARL were added to the 20 mg/L of AB25 and stirred continuously. After a period of time, the solution was filtered, and the concentration of AB25 in the filtrate was measured each time, based on the differences in AB25 concentration in the solution before and after adsorption. All adsorption studies were done in duplicate, and the results were reported in average.

3. Results and Discussions

3.1. FT-IR Analysis

A comparison of FT-IR spectra of unmodified rubber leaf powder, PARL before and after AB25 adsorption is shown in Figure 1. From Figure 1(a) and Figure 1(b), it was found that the FT-IR spectra for all the samples do not display many variations. The broad and high band ranging from 3000 to 3600 cm^{-1} indicated the overlapping of Si-OH (silanol), R-OH (hydroxyl) and $-\text{NH}_2$ (amine) stretching vibrations [9]. The sharp peaks observed at 2922 and 2850 cm^{-1} can be assigned to asymmetric and symmetric $-\text{CH}_2$ groups. The presence of carboxylic acid (RCOOH) is shown by the peaks located at 1732 and 2400-3600 cm^{-1} .

The peak at 1624 cm^{-1} corresponded to the carboxylate (COO^-) and $\text{C}=\text{C}$ stretching that can be attributed to the aromatic rings of lignin. The peaks appeared from 1400 to 1600 cm^{-1} were assigned to aromatic skeletal vibration. The peaks at 1063 and 1164 cm^{-1} were assigned to alcoholic C-O and C-N stretching vibrations, respectively; while peaks around 1150-1250 cm^{-1} corresponded to phenolic hydroxyl groups in lignin. The strong peak observed at 1060-1150 cm^{-1} could also represent C-O-C of ether group. The region below 1000 cm^{-1} is called "fingerprint region" and the adsorption cannot be clearly assigned to any particular vibration because they correspond to complex interacting wave systems [9].

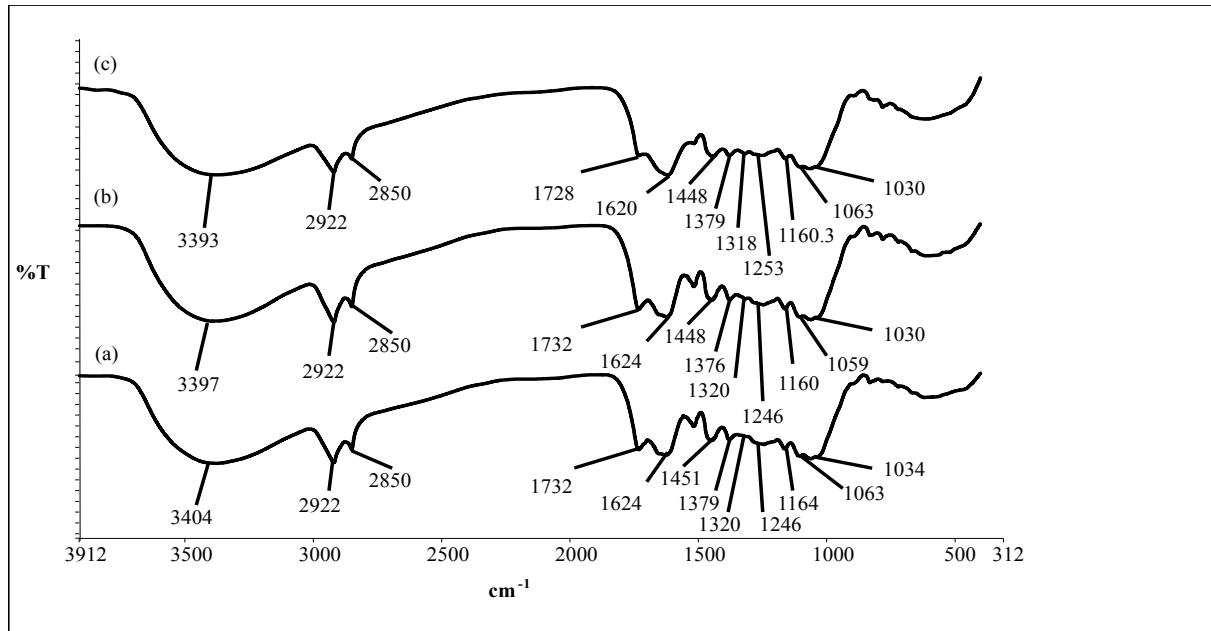


Figure 1. FT-IR spectra of (a) unmodified rubber leaf powder, (b) PARL before AB25 adsorption and (c) PARL after AB25 adsorption

The FT-IR spectrum of PARL after adsorption (Figure. 1c) shows some shift in wavenumbers. For instance, the wavenumber at 3404 cm^{-1} in untreated rubber leaf powder had shifted to 3397 cm^{-1} after phosphoric acid treatment and further shifted to 3393 cm^{-1} after AB25 adsorption, which suggests chemical interaction between AB25 and $-\text{OH}$ and $-\text{NH}$ groups. The carboxylate group were also involved in AB25 adsorption as the wavenumber shifted from 1620 to 1624 cm^{-1} . Based on the variation in the wavenumbers as indicated by the FT-IR spectra, functional groups of $-\text{OH}$, $-\text{NH}_2$, $-\text{COOH}$, $\text{C}=\text{C}$ and $-\text{COO}-$ could act as important adsorption sites for AB25 dye solution.

3.2. Effect of Initial pH

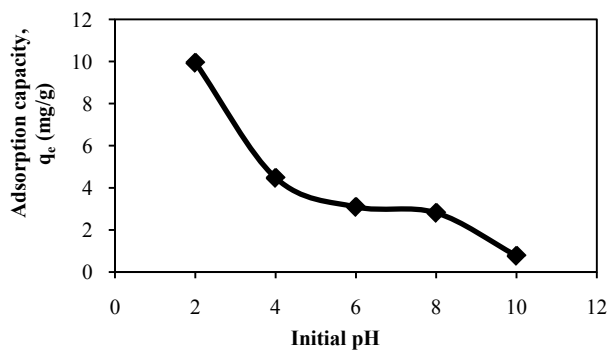


Figure 2. Effect of initial pH on the adsorption of AB25 onto PARL

Effect of pH on adsorption of AB25 dye onto PARL is shown in Figure 2. From Figure 2, pH 2.0 was found to be the optimum pH of adsorption for AB25 dye onto PARL. The adsorption capacity decreased with the increase in pH. PARL had maximum dye removal at 99.34 % at pH 2 and

dropped to 7.68 % at pH 10. As the pH increased, the number of positively charged adsorbent surface would be reduced. This phenomenon would not favour the adsorption of AB25 due to the reduction in the electrostatic attractions [2] and is recognized as the important role played by solution pH towards the dye removal onto PARL.

3.3. Effect of Agitation Time and Initial Dye Concentration

Effect of agitation time on adsorption of AB25 dye onto PARL is shown in Figure 3. The adsorption capacity increased with the increased in agitation time until equilibrium or optimum time was achieved. From the graph of adsorption capacity against agitation time, after the optimum time was reached, further agitation time did not increase the adsorption capacity. This condition showed that the process or system is in an equilibrium state. Hence, the optimum agitation time for the adsorption of AB25 dye onto PARL is 120 min.

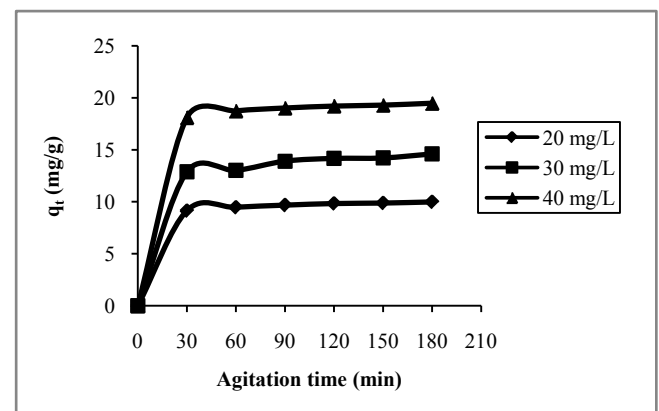


Figure 3. Effect of agitation time on the adsorption of AB25 onto PARL

The influence of the initial concentration of AB25 dye in the solutions on the rate of adsorption on PARL was also studied. The experiment was carried out at fixed adsorbents doses and varies in initial dye concentration 10, 20 and 30 mg/L at different time interval. The adsorption capacity increased with the increase in initial dye concentration. The increase in the amount of adsorption with increasing concentration was due to a higher probability of collision between adsorbate-adsorbent surfaces [10].

3.4. Effect of Adsorbent Dosage

The amount of dye removed by adsorption on PARL is presented in Figure 4. The dosage of adsorbent varied from 0.01 to 0.10 g at fixed pH and adsorbate concentration. From Figure 4, it was found that the percentage removal of the dye had increased from 69.5 to 99.4 % with the increased in adsorbent dosages. There was a decrease in amount of AB25 adsorbed with increased of the mass of adsorbents. According to Rajeev *et al.* (2014) [11], as the adsorbent dosage increases, the adsorbent sites available for the molecules also increases and consequently better adsorption takes place. Hence, the increase in the amount adsorbed with the dosage can be attributed to the increase in surface area and availability of more adsorption sites. From the graph, the amount of AB25 adsorbed decreased from 69.48 to 9.94 mg/g as the dosage was increased from 0.01 to 0.10 g using 50 mL of 20 mg/L AB25 in the solution. This condition may be due to the overlapping of adsorption sites as a result of overcrowding of adsorbents particles [12].

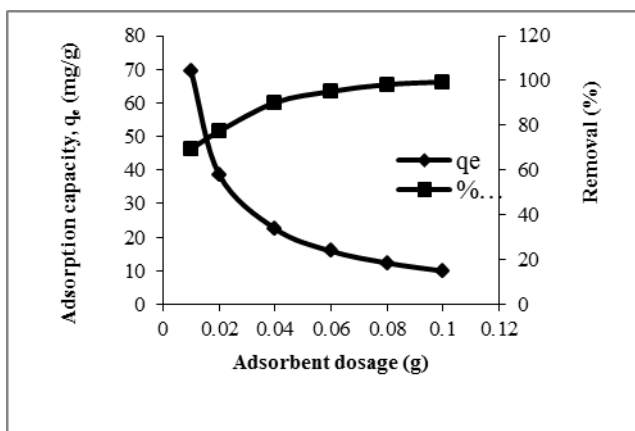


Figure 4. Effect of adsorbent dosage on the adsorption of AB25 onto PARL

3.5. Adsorption Equilibrium Isotherms

An adsorption isotherm is an expression that shows the relationship between the amounts of adsorbate adsorbed per unit weight of adsorbent (q_e , mg/g) and the concentration of adsorbate in the bulk solution (C_e , mg/L) under equilibrium conditions. The general isotherm plot as in Figure 5 shows the adsorption capacity is increased as the temperature increased.

The Langmuir and Freundlich models are often used to describe equilibrium adsorption isotherms. The Langmuir

isotherm usually indicates individual chemical adsorption (chemisorption) and reflects a relatively high affinity for adsorbate and the adsorbent within a low concentration range [13]. The most widely used Langmuir equation, which is applicable for monolayer sorption onto a surface with finite number of identical sites is indicated by:

$$C_e/q_e = 1/q_{\max} b + C_e/q_{\max} \quad (3)$$

where q_{\max} is the maximum adsorption of monolayer (mg/g), C_e is the equilibrium concentration of AB25 (mg/L), q_e is the amount of AB25 adsorbed per unit weight of PARL at equilibrium concentration (mg/g) and b is the Langmuir constant related to the affinity of binding sites (mL/mg) and also a measure of the energy of adsorption. A linearized plot of C_e/q_e against C_e gives q_{\max} and b . The Langmuir isotherm also assumes that the adsorbed layer is one molecule in thickness and that all adsorption sites have equal energies and enthalpies of adsorption. From the gradient and y-intercept of Langmuir isotherm, the values of Langmuir constant and maximum adsorption capacity for all three sets of temperatures can be determined.

Freundlich isotherm model is an empirical isotherm for non-ideal adsorption on heterogeneous surfaces as well as multilayer adsorption and is expressed as [14]:

$$\log q_e = \log K_F + 1/n \log C_e \quad (4)$$

where K_F and n are Freundlich constant that indicates adsorption capacity (mg/g) and intensity, respectively. K_F and n can be obtained from a linear plot of $\log q_e$ against $\log C_e$.

The linearized Langmuir and Freundlich models plot at 300, 310 and 320K (not shown) enabled each of the values obtained for the Langmuir and Freundlich isotherms constants and correlation coefficients to be determined (R^2), and are represented in Table 1. From Table 1, it shows that the adsorption of AB25 onto PARL is better fit to Langmuir isotherm with good R^2 values and the calculated adsorption capacity ($q_{e, \text{calc}}$) values were in agreement with the experimental adsorption capacity ($q_{e, \text{exp}}$) values at the set of temperatures. This revealed that PARL had homogeneous adsorption sites.

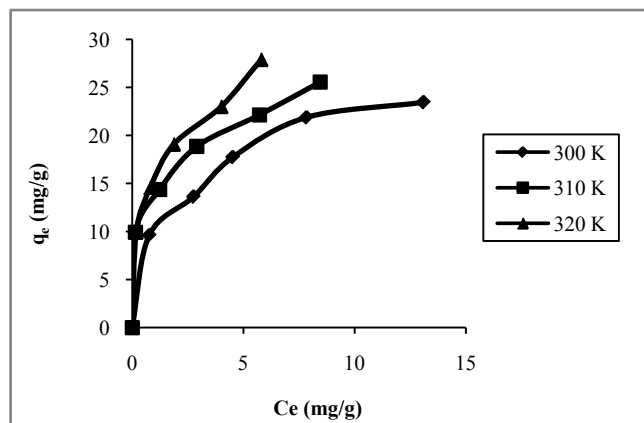


Figure 5. Adsorption isotherm of AB25 dye solution onto PARL at 300, 310 and 320 K

Table 1. Langmuir and Freundlich Isotherm Constants and Correlation Coefficients at Different Temperatures

Temp (K)	$q_{\max \text{ exp}}$ (mg/g)	Langmuir			Freundlich		
		$q_{\max \text{ cal}}$ (mg/g)	B (L/mg)	R^2	K_F (mg/g)	n	R^2
300	23.45	24.94	0.822	0.9872	10.36	3.034	0.9921
310	22.55	25.71	1.818	0.9890	14.78	4.320	0.9904
320	27.90	28.09	2.058	0.9847	15.92	3.418	0.9962

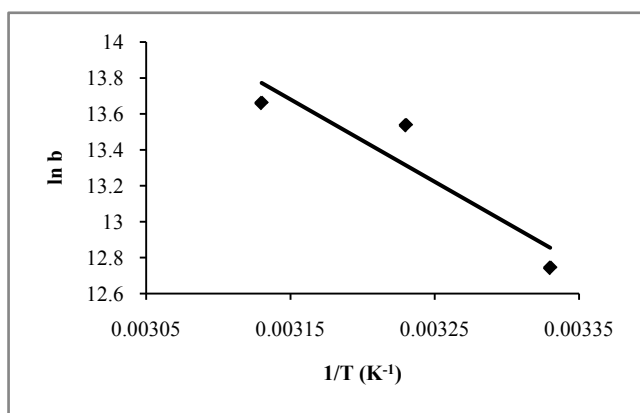
3.6. Adsorption Thermodynamics

In order to determine the spontaneity of a process of adsorption, three main thermodynamic parameters were used, namely enthalpy change (ΔH°), entropy change (ΔS°) and Gibbs free energy change (ΔG°) to test the experimental data obtained. All the thermodynamics parameters, such as ΔH° , ΔS° and ΔG° can be calculated from the following equations [8]:

$$\Delta G^\circ = -RT \ln b \quad (5)$$

$$\ln b = \Delta S^\circ/R - \Delta H^\circ/R \quad (6)$$

where b is the Langmuir constant (L mol^{-1}), R is the gas constant ($8.3142 \text{ J K}^{-1} \text{ mol}^{-1}$), and T is the temperature in kelvin (K). The slope and intercept of $\ln b$ versus $1/T$ (Figure 6), were used to calculate the values of ΔH° and ΔS° . The observed thermodynamic values are given in Table 2. The negative values of ΔG° at all temperatures indicated that the adsorption process was spontaneous, and AB25 had an excellent affinity for PARL surface. The values of ΔG° increase with increasing temperature, indicating that adsorption of AB25 onto PARL was more favourable at higher temperatures. The positive values of ΔH° showed the endothermic nature of adsorption, which means that greater adsorption of AB25 took places at higher temperatures. The positive value of ΔS° indicated an increase in randomness at the solid/solution interface after AB25 adsorption onto PARL surface.

**Figure 6.** Van't Hoff plot of AB25 adsorption onto PARL**Table 2.** Thermodynamics Parameters of AB25 Adsorption onto PARL

Temperature (K)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/K mol)
300	-31.78	38.16	233.94
310	-34.89		
320	-36.34		

3.7. Adsorption Kinetics

Pseudo-first and pseudo-second model are used to explain the mechanism of adsorption. Kinetic adsorption data were first treated with pseudo-first order kinetic model. The linear form of pseudo-first order rate equation is expressed as Equation 7 [15]:

$$\log (q_e - q_t) = \log q_e - (k_1/2.303) t \quad (7)$$

where q_e and q_t (mg/g) are the amount of AB25 absorbed onto adsorbent at equilibrium and at time t (min), respectively and k_1 (min^{-1}) is the rate constant of pseudo-first order adsorption. The straight line plots of $\log (q_e - q_t)$ against t will yield the values of the rate constant, k_1 and q_e from the slope and intercept, respectively. Figure 7 represents the pseudo-first order kinetic plot for AB25 adsorption on PARL at 300 K.

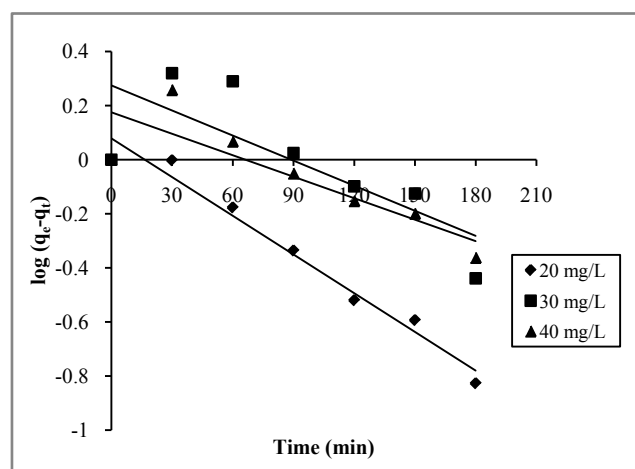
**Figure 7.** Pseudo-first order kinetic reaction of AB25 adsorption onto PARL at 20, 30, and 40 mg/L

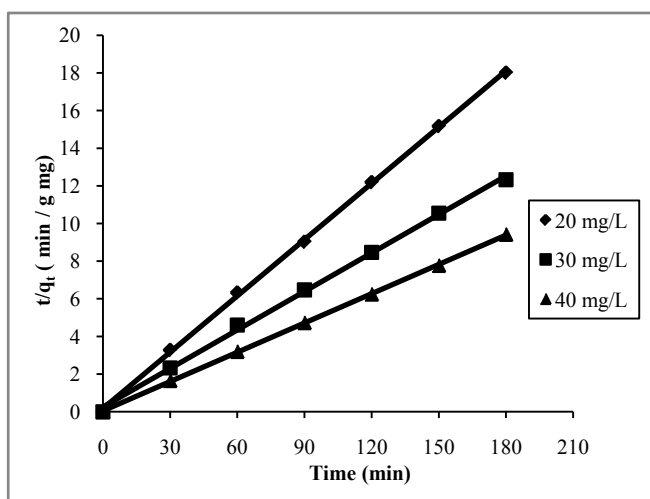
Table 3. Kinetics Parameters of AB25 Adsorption onto PARL based on Pseudo-First and Second Order Kinetic Model

AB25 (mg/L)	$q_{e, \text{exp}}$ (mg/g)	Pseudo-First Order			Pseudo-Second Order		
		k_1 (1/min)	R^2	$q_{e, \text{calc}}$ (mg/g)	k_2 (g/mg min)	R^2	$q_{e, \text{calc}}$ (mg/g)
20	10.14	0.011	0.973	1.20	0.060	0.9996	10.04
30	14.97	0.007	0.594	1.88	0.018	0.9984	14.66
40	19.91	0.006	0.734	1.50	0.050	0.9998	19.31

The pseudo-second order equation is based on the assumption that chemisorption is the rate determining step and is expressed as given by Equation 8 [16]:

$$t/q_t = 1/h + t/q_e \quad (8)$$

where $h = k_2 q_e^2$ can be regarded as the initial adsorption rate (mg/g min) as t approaches to zero, and k_2 (g/min mg) is the rate constant of pseudo-second order adsorption. The straight line plots of t/q_t against t were used to determine the rate constant, k_2 and correlation coefficient, R^2 . Figure 8 represents the pseudo-second order plot for PARL at 300 K.

**Figure 8.** Pseudo-second order kinetic reaction of AB25 adsorption onto PARL at 20, 30 and 40 mg/L

The kinetic parameters of the pseudo-first order and pseudo-second order kinetic models for adsorption of AB25 based on Figures 7 and 8, are summarized in Table 3. From the R^2 values shown, it can be concluded that the pseudo-second order kinetic model can be used to represent the adsorption behaviour over the whole range of contact time. Moreover, the calculated adsorption capacity ($q_{e, \text{calc}}$) values were in agreement with the experimental adsorption capacity ($q_{e, \text{exp}}$) value. These also revealed that the uptake of the AB25 dye onto PARL was more rapid and favourable. The initial adsorption rate, h , of the pseudo-second order kinetic model, increased as concentrations increased.

The large number of adsorption sites available on PARL might cause the rapid adsorption of AB25 at higher concentrations, possibly due to the increase in the driving

force for mass transfer, and thus, allowing AB25 to reach the adsorbent surface in a shorter period.

This study has demonstrated some vital evidences in relation to the adsorption behaviour of AB25 dye onto PARL. The implication of this finding reckons the prospect of PARL to be employed as an alternative adsorbent for a much costly activated carbon for remediation of wastewater contaminated by AB25 dye in the future.

4. Conclusions

PARL showed good adsorption capacity of AB25 dye solution. Adsorption capacity was affected by low pH and higher adsorbent dosage as well as increased in concentration and contact time. AB25 adsorption onto PARL obeyed the pseudo-second order kinetic model while equilibrium data presented a better fit to the Langmuir, isotherm model. The FT-IR analysis revealed -OH, -NH₂, -COOH, C=C and -COO- as the major functional groups that act as adsorption sites for AB25 dye solution. Hence, it can be concluded that PARL adsorbents is effective for the uptake of AB25 dye.

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