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Equilibrium studies for Basic blue 3 adsorption onto durian peel (*Durio zibethinus* Murray)

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ABSTRACT

A waste material with practically no cost, durian peel (DP) was studied for its ability to remove Basic blue 3 (BB3) from aqueous solutions. Batch kinetic studies were carried out to study the adsorption characteristics under various experimental conditions. The optimum pH for the dye removal occurred in the pH range of 3–10. Adsorption was found to be concentration dependent. The kinetics of dye adsorption fitted a pseudo-second order rate expression. Both Langmuir and Freundlich models appeared to provide reasonable fittings for the adsorption data of BB3 on DP. Application of Langmuir isotherm indicated that under the experimental condition of room temperature (25 ± 2°C) with natural pH of the dye solution and shaken at 150 rpm, the maximum adsorption capacity of DP for BB3 was 49.50 mg g^{-1}. The percentage uptake of BB3 increased with the increase in temperature. Adsorption of BB3 was found to be endothermic and various thermodynamic parameters were evaluated using van't Hoff plot.

Keywords: Durian peel; Low cost sorbent; Batch study; Adsorption; Basic blue 3; Kinetics

1. Introduction

The discharge of dyes into aquatic ecosystems has become a matter of concern over the last few decades. At present there are more than 100,000 commercial dyes with a rough estimated production of 7 × 10^5 tons per year [1]. The extensive usage of dyes in many industries to color their products resulted in a huge amount of colored waste and this is worrying for both toxicology and esthetical reasons. Therefore, the removal of dyes from wastewater has become environmentally important not only for their high chemical and biological oxygen demand and suspended solids but also for their perturbing effect in photosynthetic activity in aquatic life due to reduced light penetration. Some researchers have also determined that some dyes, dye precursors and their biotransformation products are toxic, mutagenic and carcinogenic in nature [2]. For example, basic dyes, which are predominantly used in colouring acrylic fiber are generally more toxic than other classes of dyes [3]. Thus, the removal of basic dyes (such as BB3 in this study) from the environmental is worthwhile noting.

The range of conventional methods for dye removal includes advanced oxidation process, membrane separation, ion-exchange, photodegradation and carbon adsorption. However, treating coloured wastewater by

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conventional treatment methods posted several difficulties and therefore, researches have been focused on seeking a better and economical alternative. Amongst all, liquid phase adsorption is one of the most popular investigated methods for the removal of dyes. Adsorption techniques have become increasingly popular not only because of its relatively low initial treatment cost, ease of operation, flexibility and simplicity of design, but also insensitivity to toxic pollutants and the availability of wide range of adsorbents.

The commercially available activated carbon is considered to be an efficient adsorbent for the removal of various dyes but the usage of activated carbon has been limited by its high cost due [4]. However, this adsorption process still remains as an attractive alternative for the treatment of dye wastewaters if the adsorbent is inexpensive and does not require an additional pretreatment step before its application.

Due to this reason, various plant wastes have been studied for their adsorption capacities as they are inexpensive or low-cost, abundant in nature and require little processing. Some dye-adsorbent systems which have demonstrated commercial potential include sun flower seed, Neem leaf powder, orange peel, jackfruit peel, groundnut shell, banana pith, hazelnut shell, rice husk, wood shaving, corncorb and sugarcane bagasse [5–14].

The amount of durian shell generated is approximately 350,000 tons, and direct discharge of this solid waste will cause the environmental problems [15]. Exploring the possibility usage of this locally abundant waste material not only will reduce the environment burden but could also be beneficial to the economy. However, a very limited study in the literature [16–18], has, hitherto reported on the potential of durian peel as the adsorbent for dyes removal. Therefore, in this study efforts are attempted to evaluate the feasibility of using this solid waste as an adsorbent for basic dye removal.

2. Materials and methods

2.1. Sorbent

Durian peel (DP) was collected and washed thoroughly to ensure the removal of dirt and other impurities. It was then rinsed several times with distilled water and cut into small pieces. DP was sun dried to completely remove the moisture content and the dried DP was ground to pass through a 1 mm-sieve and labelled as DP.

2.2. Sorbate

Synthetic dye solution of BB3 (C.I. = 51,004, FW = 359.89, λ_{max} = 654 nm, Aldrich 25% purity) was used as adsorbate in this study. This commercial dye was used without any further purification and the structure is shown in Fig. 1. Standard dye solution of 1000 mg l^{-1} of BB3 was prepared as stock solution and subsequently diluted when necessary.

2.3. Surface characterization

Field emission scanning electron microscopy (FESEM) analysis was carried out to study the surface morphology of DP. The micrograph was taken using FESEM JSM 6701F (JEOL) operated at emission current of 2.00 kV with working distance of 6.0 mm. The functional groups DP were determined using Perkin-Elmer System 2000 FT-IR Spectrometer. The sample disk was prepared by mixing the dried biosorbent with KBr. The mixture was then ground and compressed into a pellet before it was analyzed to obtain the spectrum.

2.4. Batch adsorption studies

All the batch adsorption experiments were performed in duplicate by agitating 0.10 g of sorbent in 20 ml of dye solution in a centrifuge tube and shaken at 150 rpm on an orbital shaker at room temperature (25 ± 2°C) unless otherwise stated. The results presented are the means value. Control without sorbent were simultaneously carried out to demonstrate dye uptake was due to sorbent and not the wall of the centrifuge tube. At the end of the adsorption process, the mixtures were centrifuged at 3000 rpm phase separation. The BB3 dye concentrations were analysed using Perkin Elmer Lamda 35 double-beam UV/visible spectrophotometer with 1.0 cm light path cuvette (quartz cell) at the wavelength corresponding to maximum absorption, λ_{max} = 654 nm. Dilutions were carried out when the measurement exceeded the linearity of the calibration curve.

The percentage uptake of BB3 was calculated by the following equation:

$$\text{Percentage removal} = \frac{C_o - C_e}{C_o} \times 100\,\% \tag{1}$$

where C_o and C_e (mg l^{-1}) are the initial and equilibrium liquid phase concentration of BB3, respectively.

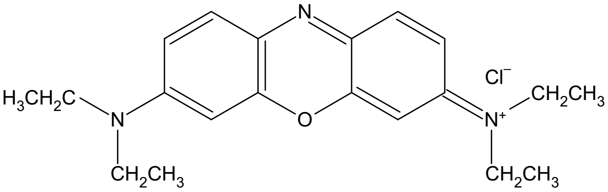

Fig. 1. The structure of BB3.

2.4.1. Effect of pH

The effect of pH in the removal of BB3 was analyzed over the range from 2 to 10. The initial pH of the dye solutions was adjusted by adding dilute HCI or NaOH.

2.4.2. Effect initial concentration and contact time

The contact time experiments were performed with concentrations ranging from 50 to 200 mg l^{-1}. The samples were withdrawn and analyzed for their dye concentrations at predetermined intervals.

2.4.3. Effect of temperature

In the effect of temperature, the experiment was carried out in a water bath shaker at the temperatures ranging from 30 to 80°C.

2.4.4. Sorption isotherm

Adsorption isotherms were obtained by varying BB3 concentrations from 25 to 200 mg l^{-1}. Linear regression analyses were also carried out for isotherm studies to identify the best fit model for the adsorption studies.

3. Results and discussion

3.1. Characterization of durian peel

Figs. 2(a) and (b) depict the FESEM images of pristine DP. An unsmooth surface with structure of irregular in shape was found in the sample prepared. As shown in the FESEM image of higher magnification (Fig. 2(b)), the DP has a non porous configuration, in well agreement with previous report [19].

The spectrum of DP was measured within the range of 4000–400 cm^{-1} wavenumber. The FTIR spectrum of DP (Fig. 3) displayed a number of absorption peaks, indicating the complex nature of the studied sorbent. Strong absorption observed in the region 3600–3200 cm^{-1} is most probably due to the hydroxyl groups of DH and moisture absorbed during the preparation of KBr disk. The peak around 1637 cm^{-1} can be assigned to the C=O stretching from the lignin carbonyl groups. The absorption peak observed at 1059 cm^{-1} corresponds to the C–O band which is also an indicative of the presence of lignin structure in DP. The additional peak at 592 cm^{-1} can be assigned to bending modes of aromatic compounds [20–22].

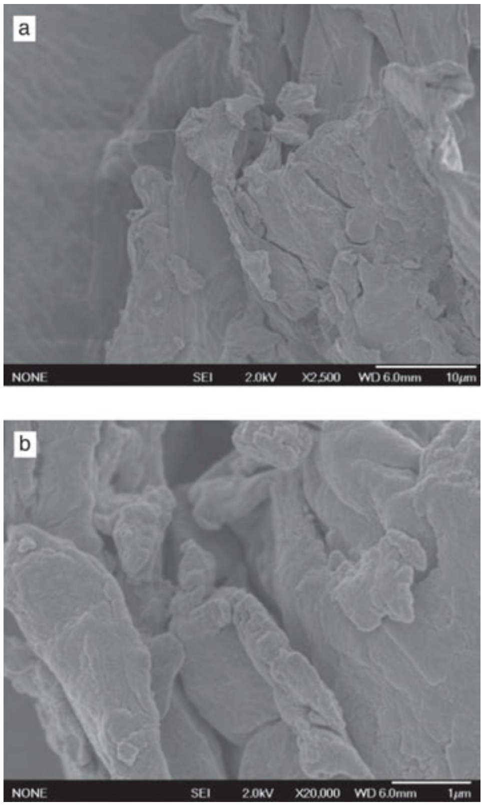

Fig. 2. FESEM images of DP with magnifications of (a) 2500 × and (b) 20,000 ×.

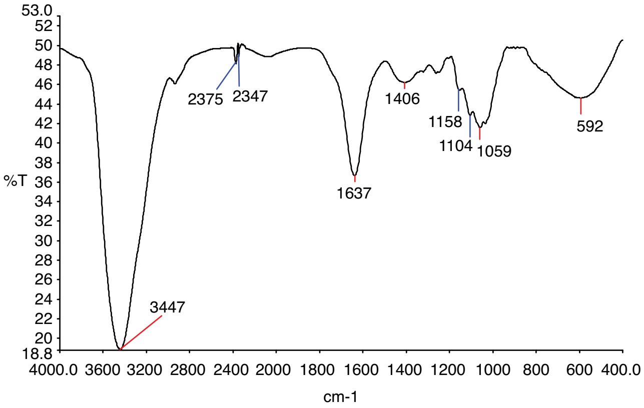

Fig. 3. FT-IR spectrum of DP.

3.2. Effect of pH

The pH at point of zero charge (pH_{pzc}) of DP was determined by the mass titration method [23] and found to be 5.0. The uptake below pH_{pzc} can perhaps be attributed by the simultaneous occurrence of different kinds of interactions such as entrapment, ion-exchange, hydrophobic attraction, physical adsorption and etc. Similar adsorption pattern has been reported in the uptake of cadmium ions by various fruit peel wastes [24].

As the surface charge of the adsorbent and the degree of ionization of the adsorbate will be affected by the pH of the solution, therefore the influence of initial pH was the first parameter to be investigated in this study. Fig. 4 shows the results of the percentage uptake of BB3 from pH 2 to 10. The percentage uptake of BB3 was lowest at pH 2 and increased sharply by 17.03% at pH 3. Thereafter, the percentage uptake became almost consistent which is in the range of 92–94%. It is suggested that at lower pH, the adsorbent's surface may get positively charged which is not favorable for the adsorption of dye cations due to electrostatic repulsion. With increasing pH, the number of negatively charged sites increased, resulting in adsorption sites that were made available for binding with BB3, and thus facilitate a higher percentage uptake. A noticeable and abrupt decrease in the percentage uptake was observed as the initial pH was increased from 10 to 11. The decrease is considered to be closely related to the precipitation that occurred. Similar observation was reported in the removal of Basic Green 4 by modified rice straw [25].

The extent of BB3 removal involving DP shows that it is strongly dependent on solution pH. Due to the presence of different functional groups on DP and its specific characteristics, it is quite possible that at least some of these mechanisms (e.g., complexation, ion-exchange due to surface ionization, and formation of hydrogen bonds) are to varying degrees acting simultaneously for the binding with BB3.

3.3. Effect of initial dye concentration and contact time

The influence of initial BB3 dye concentrations on the dye uptake by DP as a function of time is shown in Fig. 5. The rate of adsorption was very rapid and equilibrium was achieved within 120 min. The rapid uptake at the initial stage indicates that there were many available sites for adsorption and attributes to the rapid attachment of the dye molecules to the surface of the adsorbent.

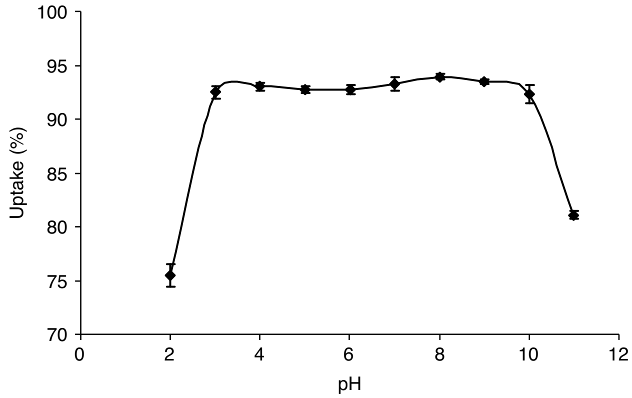

Fig. 4. Effect of pH on the adsorption of BB3 by DP (0.10 g DP; 20 ml dye solution; 150 rpm; 4 h).

It is generally accepted that in a dynamic well stirred solid-liquid system, the adsorption process can be separated into three stages [26–28]. At the first stage, BB3 dye molecules had to diffuse through the solution to the external surface of the DP, also known as film mass transfer. The second stage involved the diffusion within the pores of DP internal structure which required a longer time before it reached equilibrium and the third stage is the adsorption of BB3 on the active sites on the internal surface of the pores. In addition, it was observed that the time profile of the removal was a smooth and continuous curve leading to saturation, indicating the possibility of the formation of monolayer coverage of dye on the surface of DP [29].

3.4. Kinetic studies

In order to explore the kinetics involved in BB3 adsorption, the experimental data was studied with respect to two different kinetic models, namely pseudo-first order model [30] and pseudo-second order model [31]. The equations are expressed as follows:

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

(pseudo-first order equation) (2)

and

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e}$$

(pseudo-second order equation) (3)

where q_e is the amount of BB3 adsorbed at equilibrium (mg g^{-1}), q_t is the amount of BB3 adsorbed at time t (mg g^{-1}), k_1 is the rate constant of pseudo-first order kinetics (min^{-1}), h ($k_2 q_e^2$) is the initial adsorption rate

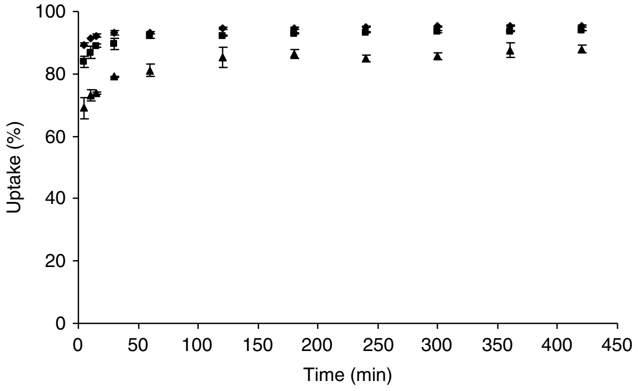

Fig. 5. Effect of initial dye concentrations and contact time on adsorption of BB3 by DP. ♦, ■, ▲ – 50, 100 and 200 mg l^{-1} of BB3 (0.10 g DP; 20 ml; natural pH of dye solution; 150 rpm).

(mg g^{-1} min^{-1}) and k_2 is the rate constant of pseudo-second order kinetics (g mg^{-1} min^{-1}).

Values of k_1 for the pseudo-first order kinetic model were obtained from the slopes of the linear plots of log ($q_e - q_t$) versus t for the studied concentrations. It was found that the pseudo first order kinetic model did not fit well for the whole range of concentrations studied as the equilibrium adsorption capacities calculated from this kinetic model gave unreasonable values compared to those determined experimentally (Table 1). The application of pseudo second order kinetic model appeared to provide a better correlation of the experimental data than the pseudo first order model for the dye system. In addition the correlation coefficients for the pseudo first order kinetic model obtained at various concentrations are in general lower than those of the pseudo second order model (Table 1).

The linear plots of pseudo-second order kinetics model with R^2 values higher than 0.99 indicates that the system under study is more appropriately described by the pseudo-second order model which was based on the assumption that the rate limiting step may be chemical adsorption or chemisorption involving valency forces through sharing or exchange of electron between adsorbent and adsorbate [32]. A number of authors have also reported on the applicability of pseudo-second order kinetics for the adsorption process [33–41]. The values of q_e, k_2 and h against C_o in the corresponding linear plots of the pseudo-second order equation were regressed to obtain expressions for these values in terms of the initial dye concentration. Each of these parameters can be expressed as a function of C_o for BB3 as reported by [32]:

$$q_e = \frac{C_o}{A_q C_o + B_q} \tag{4}$$

$$k_2 = \frac{C_o}{A_k C_o + B_k} \tag{5}$$

$$h = \frac{C_o}{A_h C_o + B_h} \tag{6}$$

where A_q, B_q, A_k, B_k, A_h and B_h are constants related to the respective equations. The constant values can then be determined from the slopes and intercepts of the linear plots accordingly, which are listed in Table 2.

Thus, the generalized predictive models for BB3 adsorbed at any contact time and initial concentration within the given range with relationship of q_t, C_o and t can be represented as follows by substituting the various values in the equations mentioned earlier into Eq. (3).

The theoretical model for BB3-DP system can therefore be represented as:

$$q_t = \frac{C_o t}{0.0973C_o - 0.1913 + (0.0026C_o + 5.1126)t} \tag{7}$$

Eq. (7) was employed to represent the generalized predictive model for BB3 adsorbed at any contact time and initial dye concentration within a given range. Fig. 6 shows a typical plot of comparison between the experimental and theoretically pseudo-second order modelled time profile for BB3 adsorption by DP. It is evident that the pseudo-second order rate law provided a good prediction for the amounts of BB3 adsorbed over the studied range.

3.5. Effect of temperature and adsorption thermodynamics

The effect of temperature in the removal of BB3 by DP showed that the percentage uptake of BB3 increased gradually as the temperature increased from 30°C to 80°C.

Table 1
Pseudo-first and pseudo-second order kinetic model parameters for different initial BB3 concentrations

Initial BB3 concentration (mg l^{-1})	$q_{e, exp}$ (mg g^{-1})	Pseudo-first order kinetic model			Pseudo-second order kinetic model			
		$q_{e, cal}$ (mg g^{-1})	k_1 (min^{-1})	R^2	$q_{e, cal}$ (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	h (mg g^{-1} min^{-1})	R^2
50	8.435	0.152	-4.145×10^{-3}	0.235	8.482	1.396×10^{-1}	10.040	1.000
100	18.377	0.851	5.988×10^{-3}	0.224	18.519	4.091×10^{-2}	14.025	1.000
200	30.860	2.805	3.455×10^{-3}	0.579	32.154	8.501×10^{-3}	8.787	0.999

Table 2
Empirical parameters for predicted q_e, k and h from C_o

A_q (g mg^{-1})	B_q (g l^{-1})	A_k (mg min g^{-1})	B_k (mg^2 min g^{-1} l^{-1})	A_h (g min mg^{-1})	B_h (g min l^{-1})
2.611×10^{-3}	5.113	1.190×10^{2}	-5.442×10^{3}	9.732×10^{-2}	-0.191

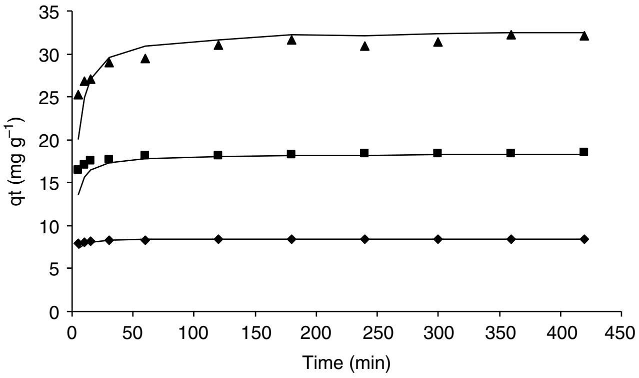

Fig. 6. Comparison between the measured and pseudo-second order modelled time profiles for BB3 adsorption by DP. ◆, ■, ▲ – 50, 100 and 200 mg l^{-1} of BB3. Symbol- experimental; Line – theoretical (0.10 g DP; 20 ml; natural pH of dye solution; 150 rpm).

This observation indicates that the adsorption process was an endothermic process and favors higher temperature. A rise in temperature increases the mobility and kinetic energy of dye molecules in solution, thus resulting in an increase in the percentage of dye uptake. This result can also be explained by the increase of the pore size and the total pore volume of DP at elevated temperature which facilitates the penetration of BB3 dye molecules into the internal structure. Alternatively, the increase of adsorption with temperature may be due to the chemisorption. Since chemisorption is mainly irreversible, the BB3 dye molecules were chemisorbed onto DP with an increasing number of molecules acquiring sufficient energy to undergo chemical reaction with the binding sites on the surface of DP. As a result, the extent of colour removal increases with elevating temperature.

The dependence of BB3 adsorption on temperature can be further confirmed by the van't Hoff plot based on the equation below:

$$\log K_{\mathrm{d}} = \frac{\Delta S^{\mathrm{o}}}{2.303R} - \frac{\Delta H^{\mathrm{o}}}{2.303RT} \tag{8}$$

where K_{d} ($q_{\mathrm{e}}/C_{\mathrm{e}}$) is the distribution coefficient, T is the absolute dye solution temperature (K) and R is the universal gas constant (8.314 J mol^{-1} K^{-1}).

A plot of log K_{d} against $1/T$ gave a linear graph as shown in Fig. 7. It can be observed that the logarithm of the distribution coefficient decreased linearly with increasing temperature. The values of $\Delta H°$ and $\Delta S°$ determined from the slope and intercept of the van't Hoff plot were 10.13 kJ mol^{-1} and 40.85 J mol^{-1} K^{-1}, respectively. The positive value of $\Delta H°$ indicates the endothermic nature of the adsorption interaction. Whereas the positive value of $\Delta S°$ shows the increase of disorder at the solid-solution interface during the adsorption of BB3 on the DP.

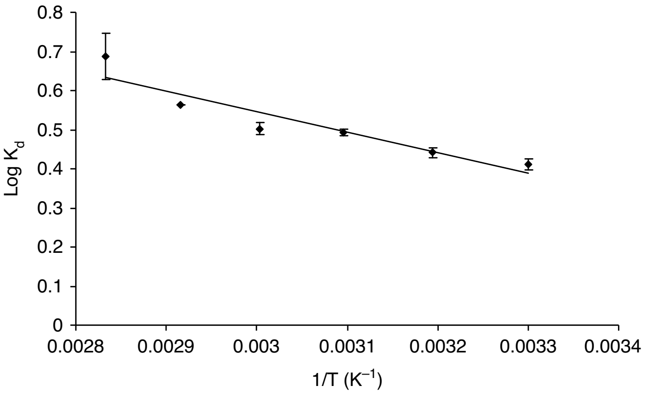

Fig. 7. van't Hoff equation plot on adsorption of BB3 by DP (0.10 g DP; 20 ml; natural pH of dye solution; 100 mg l^{-1}; 4 h;150 rpm).

The adsorption increase the randomness at the solid-solution interface with some structural changes in the adsorbate and adsorbent and an affinity of the adsorbent [42]. On the other hand, the negative values of $\Delta G°$ and the shift in magnitude of $\Delta G°$ to higher negative values at higher temperature (Table 3) are indicative of a rapid and more spontaneous adsorption at higher temperature.

3.6. Adsorption isotherms

The adsorption properties and equilibrium data, commonly known as adsorption isotherms can provide the basic and fundamental information on the adsorbate-adsorbent interactions. As the information gathered from this parameter are important for evaluating the applicability of the adsorption process as a unit operation therefore the equilibrium adsorption data of BB3 on DP are fitted into two well-known isotherms, namely Langmuir and Freundlich equations.

The linear form of Langmuir isotherm model is given by the following equation:

$$\frac{C_{\mathrm{e}}}{q_{\mathrm{e}}} = \frac{1}{Q_o K_{\mathrm{L}}} + \frac{C_{\mathrm{e}}}{Q_o} \tag{9}$$

Table 3
The change in free energy (ΔG) for the adsorption of BB3 onto DP

Temperature, T (K)	$\Delta G°$ (kJ mol^{-1})	K_{d} (l g^{-1})
303	−2.250	2.443
313	−2.659	2.778
323	−3.067	3.133
333	−3.476	3.498
343	−3.884	3.904
353	−4.293	4.318

whereas the linear form of Freundlich isotherm model can be represented as:

$$\log q_e = \frac{\log C_e}{n} + \log K_f \tag{10}$$

where C_e is the equilibrium liquid phase dye concentration (mg l^{-1}), q_e is the amount of dye adsorbed at equilibrium (mg g^{-1}), Q_o is the maximum adsorption capacity (mg g^{-1}), K_L is the adsorption equilibrium constant (l mg^{-1}), n is Freundlich constant for intensity and K_f is Freundlich constant for adsorption capacity. The slope of $1/n$ ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero [43]. A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption [44].

The parameters determined from each isotherm model are listed in Table 4 whereas the linear Langmuir and Freundlich plots of BB3-DP systems are shown in Figs. 8 and 9, respectively. Although based on different assumptions: Langmuir model implies monolayer coverage and constant adsorption energy while the Freundlich model deals with physicochemical adsorption on heterogeneous surfaces, the experimental data appeared to fit in well in both isotherm models. Applicability of both isotherms to adsorption of dyes by agricultural wastes, activated carbons prepared from wastes and treated spent bleaching earth have been reported previously [33,45–47]. Notwithstanding this, Langmuir model allows the calculation of maximum adsorption capacities that could be useful for the comparison of the adsorption efficiency of the material under studied with other adsorbents (Table 5).

Table 4
Langmuir and Freundlich isotherm model constants and correlation coefficients for sorption of BB3 by DP

Langmuir isotherm				Freundlich isotherm		
Q_o (mg g^{-1})	K_L (l mg^{-1})	R^2	R_L	K_F	$1/n$	R^2
49.50	0.092	0.995	0.056	4.854	0.666	0.984

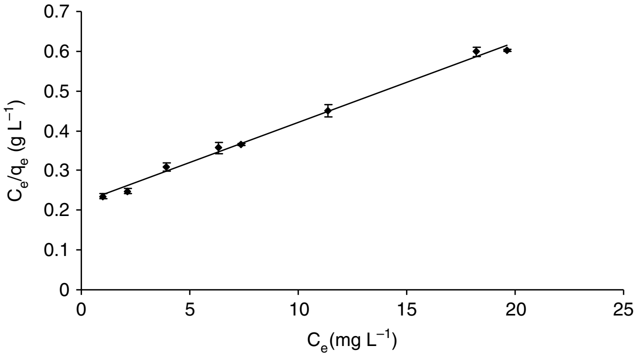

Fig. 8. Langmuir isotherm on adsorption of BB3 by DP (0.10 g DP; 20 ml; natural pH of dye solution; 4 h;150 rpm).

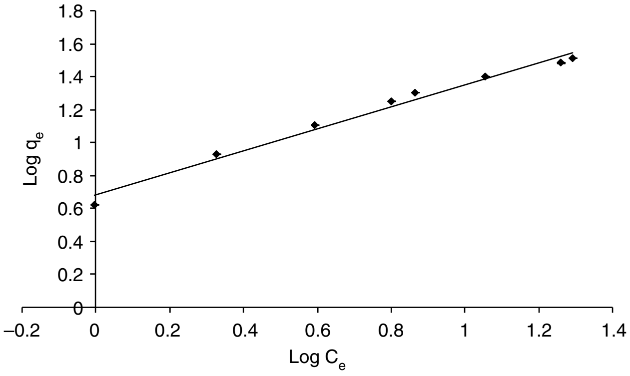

Fig. 9. Freundlich isotherm on adsorption of BB3 by DP (0.10 g DP; 20 ml; natural pH of dye solution; 4 h;150 rpm).

Table 5
Comparison of maximum adsorption capacity for BB3 by different adsorbents

Adsorbent	Maximum adsorption capacity (mg g^{-1})	Reference
Activated carbon	648.6	[48]
Activated sludge biomass	36.5	[49]
Palm fruit bunch	92	[48]
Ethylenediamine modified rice hull	3.29	[33]
Quartenised sugar cane bagasse	5.58	[50]
Corynebacterium glutamicum	7.90	[51]
NTA modified sugarcane bagasse	54.35	[34]
Natural sugarcane bagasse	23.64	[52]
Natural durian peel	49.50	This study

4. Conclusions

This study has shown the effectiveness of DP as a potential low cost adsorbent for the removal of BB3 in aqueous solutions. In the batch studies, the results indicated that adsorption capacity of DP was considerably affected by initial BB3 concentrations, contact time, pH and temperature. The optimal pH for the removal of BB3 was in the range of 3–10. Equilibrium adsorption data conform to both Langmuir and Freundlich isotherms and maximum adsorption capacity was 49.50 mg g^{-1}. Analysis of the kinetics data implied that pseudo-second order kinetics model provided a better correlation of the experimental results than pseudo-first order kinetic. The adsorption profiles derived based on the pseudo-second order kinetic model showed good agreement with the experimental curves. Besides, the percentage of BB3 uptake increased with elevating temperature and the thermodynamics data showed that the adsorption was an endothermic process.

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