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Adsorptive removal of anionic dye (Reactive Black 5) from aqueous solution using chemically modified banana peel powder: kinetic, isotherm, thermodynamic, and reusability studies

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ABSTRACT

The removal of Reactive Black 5 (RB5) using chemically modified banana peel powder (CMBPP) from aqueous solution was dealt with in the present investigation. Factors affecting the adsorption of RB5 (like pH solution, agitation speed, initial concentration of RB5, contact time and temperature) were investigated. FTIR, SEM-EDX, BET and Elemental analysis characterized the adsorbent material. Adsorption kinetic results evaluated by non-linear pseudo-second-order model was fitted well and showed good correlation with the experimental data than the pseudo-first-order model. experimental equilibrium data evaluated by non-linear Langmuir, Freundlich, The Dubinin-Radushkevich (D-R), and Temkin equations and the experimental data were well described by the Langmuir isotherm model. Langmuir monolayer sorption capacity of RB5 onto CMBPP was observed at pH 3.0 (211.8 mg/g). The values of thermodynamic parameters revealed that the sorption process was feasible, spontaneous, endothermic, and physisorption in nature, i.e. $(\Delta G^{\circ} < 0, \Delta H^{\circ} > 0, \text{ and } \Delta S^{\circ} > 0)$. Desorption studies reveal that the maximum recovery of RB5 when 0.1 M NaOH solution used as a desorbent. The CMBPP also exhibited excellent regeneration efficiency for the five cycles of successive adsorption-desorption. The results exposed that CMBPP could use as a prospective adsorbent material for the removal of RB5 from aqueous media.

KEYWORDS

Adsorption; isotherms; kinetics; Reactive Black 5; thermodynamics

Introduction

The severe concern on the environment is increased worldwide due to human activities. Water pollution is one such area that needs massive attention as it directly affects human health and other living organisms. Azo dyes occupied the primary position of concern on account of their esthetic and toxicological properties, among the different pollutants of the aquatic eco-system. The primary source of dyes in the wastewater is textile manufacturing, leather tanning, printing, cosmetic, paper, plastics, and pharmaceutical industries. The estimated dye percent in the effluent is up to 10-15% of the total dyes used. The adverse property, such as persistence, biotoxicity, and bioaccumulation of dyes, made them hazard to the eco-systems and human health (Ranjbari et al. 2019). Among different chemical structural type, (acidic, direct, reactive, basic, anthroquinone-based, disperse, diazo and metal complex) reactive azo dyes are usually used in textile industries. Simple dyeing method and covalent binding with cellulose fibers gave them such an advantage over the other dyes. Due to hydrolysis side reaction during the dyeing process, around 20% of dye goes to effluent that gives high color. Many reactive dyes cause severe damage to the central nervous and digestive systems particularly to the liver of human beings because of their mutagenic and carcinogenic nature and pollute the agricultural cultivation and underground water (El-Zawahry *et al.* 2016). Therefore, it is necessary to remove reactive dyes from effluents to make safe discharge in aqueous environments.

Reactive Black 5 is a type of diazo acidic reactive dye, and its breakdown products are carcinogenic and mutagenic; hence, its presence in water is of great concern. Therefore, care should be taken in case of effluents discharged into the aquatic environment to minimize pollution (Bhaumik *et al.* 2016). A variety of conventional wastewater treatment processes include electrolysis (Yi *et al.* 2008), chemical oxidation (Osugi *et al.* 2009), ion exchange (Labanda *et al.* 2009), biodegradation (Stolz 2001), photocatalysis (Han *et al.* 2009), advanced oxidation (Alaton *et al.* 2002) and adsorption

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(Yuvaraja *et al.* 2019) have been employed to eliminate azo dyes from industrial effluents. Among them, the adsorption process was found to be efficient, economical, can be operative at low concentrations in continuous or batch processes, comfortable performance over other conventional treatment technologies/processes, regeneration, and reuse. Using an adsorption technique, removal of RB5 from aqueous solution is the objective of this research.

Banana peels are readily available (market and household garbage), abundant and low-cost agricultural waste materials and their disposal pose a severe problem. The application of banana peels as an adsorbent solves both the issues of removal of the various pollutants from the wastewater as well as disposal of banana peels. Banana peels consist of high carbon content (because of the presence of hemicelluloses, cellulose, chlorophyll, pectin, and other low molecular weight species) hence are indeed an excellent precursor to produce activated carbon. Mainly the functional groups like hydroxyl and amine that exist on the surface of banana peel proven as suitable binders in the adsorption processes. Recent research focused on alternative biosorbents such as low-cost, natural materials, industrial solid wastes, and agricultural by-products revealed that cellulose-containing materials displayed potential sorption capacity for several types of pollutants. For the past several years, a variety of agricultural residues such as banana and orange peels (Temesgen et al. 2018), breadnut peel (Lim et al. 2017), salix babylonica (weeping willow) (Khodabandehloo et al. 2017), Prunus dulcis (Jain and Gogate 2018), peanut husk (Sadaf and Bhatti 2014), sphagnum peat moss (Hemmati et al. 2016), potato peel (Stavrinou et al. 2018), pomegranate peels (Jawad et al. 2018), mango peel (Fauzi Abdullah et al. 2016), enteromorpha sp. (Rangabhashiyam et al. 2016b), Sterculia villosa roxb. Shells (Patra et al. 2019), Swietenia mahagoni shell (Rangabhashiyam et al. 2016a), Gliricidia sepium leaf powder (Suganya et al. 2019), Trichoderma harzianum (Karthik et al. 2018), laccase modified red mud (Nadaroglu et al. 2014), silica fume (Kalkan et al. 2014), apolaccase (Nadaroglu et al. 2015), thermally modified wood (Tarmian

and Mastouri 2018), and etc. have been applied in their raw form or after some chemical modifications for the removal of dyes from wastewaters. Chemical modifications enhance the functional group potential and the number of active sites (Mohebali *et al.* 2019) thus improves their adsorption properties. Adsorption process generally depends on the adsorbate characteristics and adsorbent surface chemistry. Therefore, the role of surfactants (anionic and cationic) shows a high impact on the adsorption process (Example: cationic surfactant increases the adsorption of anionic dyes) depending on the dye type. The surfactants incline/adsorb at the surface/interface orderly as they chemically constitute a hydrophobic tail and a hydrophilic head.

In this study, we used the CMBPP a sorbent for the removal of RB5 from the aqueous environment in the batch adsorption process. Factors influencing RB5 removal efficiencies such as pH (3.0–10.0), contact time (0–420 min), agitation speed (50–40 rpm), initial concentration of RB5 (100–400 mg/L) and temperature (298–328 K) have been considered. The ability of CMBPP for RB5 adsorption was evaluated by applying kinetics (pseudo-first- and pseudo-second-order), isotherms (Langmuir, Freundlich, D–R and Temkin) and thermodynamics (ΔG° , ΔS° , and ΔH°) parameters. Also, the reusability of the CMBPP adsorption–desorption studies was carried out.

Material and methods

Chemicals and equipments

Reactive Black 5 obtained from Sigma Aldrich (India), was used as an adsorbate. The general characteristics of the RB5 are shown in Table 1. The remaining chemicals (HCl, NaOH, HCHO, and HCOOH) used without further purification were of analytical reagent grade. UV/Vis spectrophotometer (JASCO V-750, Japan) at the maximal adsorption wavelength (λ_{max}) 597 nm was used to measure the adsorption (before and after) of the RB5 samples. The digital pH

Table 1. Characteristics of Re	eactive Black 5.	
haracteristics Reactive Black 5		
IUPAC name	4-amino-5-hydroxy-3,6-bis((4-((2-(sulfooxy) ethyl)sulfonyl) phenyl)azo)-2,7-naphthalenedisulfonic acid tetrasodium salt.	
Chemical name	Remazol Black B	
Molecular formula	C ₂₆ H ₂₁ N ₅ Na ₄ O ₁₉ S ₆	
Dye content	≥ 50%	
Molecular weight	991.82 g/mol	
Wavelength	597 nm	
Nature	Anionic dye	
Structure	NaO、O	
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meter having a combined glass electrode (Elico, Hyderabad, India) was used for the pH measurement.

Preparation of RB5 solution

By dissolving an accurate weight of RB5 in double deionized water, 1000 mg/L stock was prepared, from that, different working concentrations (100 to 1000 mg/g) were made by adequate dilution. Calibration curve for the RB5 was prepared by determining the absorbance of various concentrations.

Preparation of adsorbent

From the local market, Nellore District, Andhra Pradesh, India were collected a bunch of Banana peels (BPs) and cleaned thoroughly rinsed with double de-ionized water to remove surface dust particles and other impurities. Dried at $80 \,^{\circ}$ C for 24 h in an oven and evaporated all the moisture after making peels into small pieces. Then grounded to a fine powder and sieved through a 150 &mgr;m size mesh, the obtained power was named as Banana Peel Powder (BPP).

The modification of the amino group was made according to the previously reported method (Gong *et al.* 2005). For the preparation of CMBPP, 9g dry weight of the BPP was mixed with a mixture of 180 mL of HCHO and 360 mL of HCOOH solution and was shaken a rotary shaker at 125 rpm for 6 h at 298 K. The reaction suspension was filtered and washed with double deionized water until filtrate becomes neutral. The filtrate called CMBPP was dried in an oven for 24 h at 70 °C to get constant weight. The reaction scheme occurs as follows:



Characterization

FTIR (Nicolet IS10, Thermo Scientific, Waltham, MA) spectroscopy was used for the characterization of CMBPP before and after RB5 adsorption in the frequency range of $400-4000 \text{ cm}^{-1}$. The surface morphology and elemental composition were analyzed by using an SEM-EDX (JEOL, JSM-7600F, Japan). BET adsorption–desorption isotherm studies at 77 K with a gas adsorption analyzer (BELSORP, BEL Japan) were used for determining the average pore diameter, specific surface area and total pore volume of CMBPP. Elemental analyzer (Thermo, Flash EA1112, Waltham, MA) was used for elemental analysis of the CMBPP.

Adsorption experiments

The following conditions were applied while doing the batch adsorption studies; pH (3.0-10.0), contact time (0-420 min),

initial concentration of RB5 (100-400 mg/L), agitation speed (50–400 rpm) and temperature (298–328 K). The pH (adjusted by adding 0.1 M HCl/NaOH) experiments were done by agitating 30 mL of RB5 solution (300 mg/L) having 0.05 g of CMBPP in falcon tubes (50 mL). Isotherm studies were carried out by transferring 0.05 g of CMBPP, separately into a series of falcon tubes filled with 30 mL of RB5 solution with initial concentrations of 100-1000 mg/L and a temperature of 298 K. The falcon tubes were placed in a thermostated mechanical shaker for 180 min, the agitation speed was 200 rpm other to attain equilibrium. For kinetic studies, 0.05 g of CMBPP was added with 30 mL of different RB5 initial concentrations (100, 200, 300, and 400 mg/L) and concentrations measured at appropriate time intervals. For thermodynamic tests adsorption experiments were carried out at various temperatures with an initial concentration of 300 mg/L. In the end, before determining the concentration of RB5 by UV/Vis spectrophotometer, the samples were centrifuged and filtered. Each adsorption experiment was conducted in triplicate and the average value was used in data analysis. The adsorption amount of RB5 onto CMBPP was calculated according to the following equation:

$$q_e = \frac{(C_i - C_e)V}{M} \tag{1}$$

Desorption and regeneration studies

Desorption of RB5 was performed to check the regeneration and recycling ability of CMBPP. For desorption studies, 0.1 g of CMBPP was added with 30 mL of (300 mg/L) RB5 solution (298 K, pH 3.0) and agitated for 180 min at 200 rpm. Contents were centrifuged, filtered and analyzed for RB5 concentration. RB5-loaded CMBPP was collected and gently washed to remove any unadsorbed RB5 with double deionized water. The spent adsorbents were then shaken with 30 mL of desorbents (de-ionized water, 0.1 M NaOH, 0.1 M CH₃COOH, 0.1 M HCl, 0.1 M H₂SO₄ and 0.1 M HNO₃) and the concentration of RB5 in the aqueous phase was measured. By best desorbent the adsorption–desorption studies were repeated for five times. The desorption efficiency of RB5 from CMBPP was calculated from the following equation:

Desorption efficiency =
$$\frac{\text{Amount of RB5 desorbed}}{\text{Amount of RB5 adsorbed}} \times 100$$
(2)

Error analysis

Error functions like chi-square (χ^2) and residual root mean square error (RMSE) were identified for finding the suitable (lower value of χ^2 and RMSE) isotherm that perfectly explains the observed equilibrium data, and they (χ^2 and RMSE) can be expressed as follows:

$$\chi^{2} = \sum_{i=1}^{m} \left(\frac{(q_{e} - q_{e,m})^{2}}{q_{e,m}} \right)$$
(3)

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RMSE =
$$\sqrt{\frac{1}{m-p} \sum_{i=1}^{m} (q_e - q_{e,m})^2}$$
 (4)

The χ^2 and RMSE values are the main criteria for choosing the best fitted isothermal model for the obtained experimental data.

Results and discussion

Characterization of the CMBPP

Figure 1 shows the FTIR spectra of the CMBPP before and after RB5 adsorption. The broad and strong peak at 3287 cm^{-1} , which indicates the presence of hydroxyl (–OH) and amine (–NH) groups on the surface of CMBPP (Figure 1a). Most of the amine groups were converted to N(CH₃)₂ groups. The peak observed at 2920 cm^{-1} can be attributed to the C–H stretching vibrations of the alkyl group. The strong peak at 1652 cm^{-1} can be attributed to the stretching vibration of C=O from the carboxyl group. The peak at 1577 cm^{-1} is assigned to the stretching vibrations of C–O or



Figure 1. FTIR spectra of CMBPP (a) before adsorption and (b) after RB5 adsorption.

C=C. The peak at 1487 cm^{-1} was assigned to the bending OH vibration of hydroxyl groups. The peak at 1240 cm^{-1} corresponds to the stretching vibrations of C–H. The two peaks at 1150 and 1097 cm^{-1} were assigned to C–O and C=O groups, respectively. FTIR spectra of RB5-loaded CMBPP (Figure 1b) showed that the peaks expected at 3287, 2920, 1652, 1577, 1487, 1240, 1150, and 1097 cm^{-1} had shifted, respectively, to 3272, 2965, 1637, 1525, 1442, 1225, 1167, and 1067 cm^{-1} due to RB5 adsorption. The shift in peak position represents the contribution of that functional group in the adsorption process (carboxyl, hydroxyl, and amine). Minor changes that observed in other frequencies were not considerable.

As shown in Figure 2a, the surface was rough, which indicates high porous nature as well as surface area, which are responsible for molecular diffusion and adsorption of contaminants. It can be seen from Figure 2b that after RB5 adsorption, the dye molecule occupied and the surface of adsorbent became smoother. The EDX spectrum of the CMBPP was shown in Figure 3c clearly shows that the composite mainly contains C (53.98 weight%, 60.02 atomic%), N (15.36 weight%, 19.41 atomic%), O (28.49 weight%, 19.61 atomic%), Mg (0.50 weight%, 0.27 atomic%), P (0.87 weight%, 0.38 atomic%), S (0.46 weight%, 0.19 atomic%), and K (0.35 weight% 0.12 atomic%).

The calculated average pore diameter, surface area and pore volume utilizing BET equation in the present study were $9.85 \text{ nm},118 \text{ m}^2/\text{g}$ and $0.5736 \text{ cm}^3/\text{g}$, respectively. The high surface area of CMBPP was beneficial for the effective removal of RB5 from aqueous media.

Proximate and elemental analysis of CMBPP is presented in Table 2. Elaborately the proximate percentage content of moisture, volatile, ash and fixed carbon were 14.32 ± 0.19 , 19.58 ± 0.02 , 13.02 ± 0.45 , and 53.08 ± 0.78 , respectively. Hence the carbon content of CMBPP is very high that facilitates CMBPP suitable for RB5 adsorption (Bello *et al.* 2013).

Effect of pH

One of the most critical aspects that determine the sorption process is solution pH because it influences the degree of ionization, the surface charge of the biosorbent and nature of dye solution. The effect of pH on the adsorption of RB5 onto CMBPP was studied at various pH levels (3.0-10.0). The pH of all solutions was attuned to values between 3.0 and 10.0. Figure 3a shows that the amount of RB5 adsorbed at equilibrium decreased from 120 to 4.9 mg/g when the pH of the solution was raising from 3.0 to 10.0. The maximum uptake of RB5 (120 mg/g) occurred at pH 3.0. The concentrations of H⁺ ions are more at pH 3.0; hence, by adsorbing them, adsorbent surface acquires a positive charge. The dye having an anionic nature, therefore strong electrostatic attraction exists between dye and the positively charged surface of the adsorbent resulting in a higher uptake of RB5 dye. When the pH increases the negatively charged sites also increases but there is a drop in positively charged sites, in such conditions electrostatic repulsion takes place between

the biosorbent surface and anionic dye resulted in a decrease of the uptake of RB5. In addition to that -OH ions in the medium (high pH) competes with the anionic dye towards the adsorbent surface. A similar trend was observed for the adsorption of RB5 with different adsorbents (Samarghandy *et al.* 2011; Shirzad-Siboni *et al.* 2014; Munagapati *et al.* 2018). Therefore, the optimum pH solution was fixed at 3.0 for further studies.

Effect of contact time

Contact time is one of the main parameters that profoundly influences the adsorption process. The sorption capacity of RB5 by CMBPP at different contact times (0–420 min) at different initial RB5 concentrations (100–400 mg/L) were studied and the results are illustrated in Figure 3b. It was observed that the adsorption capacity of the RB5 within the first 90 min was very fast, which remarkably improved as the initial dye concentration of RB5 was increased. Further increase in the contact time beyond 90 min yielded a slowed RB5 adsorption rate with equilibrium attained within 180 min total contact time. There was a rapid increase in the sorption capacity of RB5 in the initial stages, which became saturated after achieving equilibrium. The obtainability of unoccupied adsorption sites enabled the dye molecules to interact readily at the adsorbent surface at the initial stage, but at the equilibrium, the adsorption sites are covered by the dye and exert repulsive forces toward the incoming dye molecules (Gupta *et al.* 2013; Munagapati *et al.* 2017). Thus, the contact time of 180 min was selected as the optimum value for further studies.

Effect of agitation speed

In the sorption phenomena, agitation speed is an important parameter which regulates the outside boundary film formation and uniform distribution of solute in the bulk solution (Bharathi and Ramesh 2013). By keeping the remaining parameters constant, *i.e.*, 298 K, pH 3.0, 0.05 g of the sorbent for 30 mL of 300 mg/L initial RB5 solution the agitation speed was varied in the range of 50–400 rpm. The results were graphically represented in Figure 3c. Up to 200 rpm, the adsorption capacity for RB5 is increased with an increase in agitation speed; after that, it maintains a steady-state because, after 200 rpm the immobilized particles were found to be disrupted. This may be assumed due to either effect of acidic medium or inter collision of particles at higher agitation speed or both. Hence 200 rpm was selected for the entire study.



Figure 2. SEM images of (a) CMBPP (b) RB5-loaded CMBPP and (c) EDX spectrum of CMBPP.



Figure 3. Effect of (a) pH, (b) contact time, (c) agitation speed, and (d) temperature on the adsorption of RB5 onto CMBPP.

Table2. Physico-chemicalthe CMBPP.	characteristics of		
Parameter	CMBPP		
Proximate analysis			
Volatile (%)	19.58 ± 0.02		
Moisture content (%)	14.32 ± 0.19		
Ash content (%)	13.02 ± 0.45		
Fixed carbon (%)	53.08 ± 0.78		
Ultimate analysis			
Carbon (%)	56.94 ± 1.62		
Hydrogen (%)	7.92 ± 0.07		
Nitrogen (%)	0.46 ± 0.01		
Sulfur (%)	0.06 ± 0.01		
Oxygen (%)	34.62 ± 1.19		

Adsorption kinetics

To define the adsorption efficiency and solute uptake rate adsorption kinetic experiments were conducted. Kinetic models in the non-linear form were used to fit the experimental data to explain the underlined process. The pseudo-first- and pseudo-second-order non-linear kinetic equations can be described as following:

Pseudo-first-order model (Lagergren 1898):

$$q_t = q_{e1}(1 - \exp(-k_1 t))$$
 (5)

Pseudo-second-order model (Ho and McKay 1999):

$$q_t = \frac{q_{e2}^2 k_2 t}{1 + q_{e2} k_2 t} \tag{6}$$

The experimental values of RB5 uptake (q_e) , predicted RB5 uptake $(q_{e1} \text{ and } q_{e2})$, rate constants $(k_1 \text{ and } k_2)$ and correlation coefficients (R^2) are presented in Table 3. From Table 3, the results show that experimental and predicted sorption capacity increases with a raise in dye concentration. The kinetic data is well fitted with the pseudo-second-order model to explain the adsorption of RB5 onto CMBPP. This is explained by observing the R^2 values for pseudo-second-order (0.9840-0.9958) and pseudo-first-order (0.9634-0.9795) models where the former one is higher and closer to one. Also, the experimental and predicted q_e values are in good agreement. The adsorption kinetic curves of RB5 with different concentrations are shown in Figure 4.

Adsorption isotherm studies

The adsorption process is analyzed by the adsorption isotherm method to define the interaction between adsorbent and adsorbate, which is very crucial. The isotherm data of RB5 adsorption onto CMBPP were fitted using four

Table 3. Non-linear kinetic parameters for the adsorption of RB5 onto CMBPP.

			Pseudo-first-order model			Pseudo-second-order model		
Adsorbent	Conc. (mg/L)	$q_{e, \exp}$ (mg/g)	q _{e1,cal} (mg/g)	k ₁ (1/min)	R ²	$q_{e2,cal}$ (mg/g)	k_2 (g/mg min)	R ²
СМВРР	100	59	72.6	0.0174	0.9795	59.8	0.0002	0.9854
	200	92.8	112.9	0.0201	0.9634	94.4	0.0002	0.9840
	300	123.2	142.1	0.0272	0.9767	122.9	0.0003	0.9938
	400	145.7	166.3	0.0284	0.9779	144.8	0.0005	0.9958



Figure 4. Non-linear pseudo-first-order and pseudo-second-order plots for the adsorption of RB5 onto CMBPP.

non-linear isotherm equations, namely Langmuir, Freundlich, D–R, and Temkin (Munagapati *et al.* 2019).

The non-linear form of the Langmuir isotherm model is represented by Eq. (7) as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{7}$$

The hypothesis behind the Langmuir isotherm is sorption process occurs only once at precise homogeneous sites present on the adsorbent surface and further adsorption could not take place at that particular site, as full coverage has occurred. For the current study, the Langmuir adsorption capacity (q_{max}) is 211.8 mg/g and Langmuir constant is 0.0075 L/mg.

The non-linear Freundlich isotherm model is given as Eq. (8), is based on the assumption that adsorbent has heterogeneous surface sites for adsorption with varying energy levels.

$$q_e = K_f C_e^{1/n} \tag{8}$$

The magnitude of *n* value demonstrates the favorability of the adsorption process; specifically, when the value of *n* lies in 1 < n < 10 range. The value of *n* for RB5 (3.004±0.405) which lie in favorable limit indicated that the adsorption of RB5 onto CMBPP favors physiosorption.

D-R isotherm describes the pore-filling mechanism for the sorption process, which can be generally described on

both type (homogeneous and heterogeneous) of surfaces. The non-linear form of D-R isotherm model is demonstrated as the following equation:

$$q_e = q_m \exp\left(-K\varepsilon^2\right) \tag{9}$$

The Polanyi potential (ε) was calculated using the following relation:

$$\varepsilon = RT\ln\left(1 + \frac{1}{C_e}\right) \tag{10}$$

The mean free energy of adsorption, E (kJ/mol) is derived from the following equation:

$$E = \frac{1}{\sqrt{2K}} \tag{11}$$

To differentiate between physical and chemical sorption the 'E' per molecule of adsorbate is utilized (Munagapati *et al.* 2017). If the value of E < 8 kJ/mol, it implies physical sorption and E > 16 kJ/mol signifies chemisorptions process whereas 16 > E < 8 kJ/mol, means the process is ionexchange. For the present study, the mean free energy value (*E*) calculated is 2.8 kJ/mol, indicates that the mechanism involved in RB5 adsorption by CMBPP is a physiosorption process due to electrostatic or Van der Walls's attractions.

Temkin isotherm model assumed that an equal sharing of binding energies specifies the heat of adsorption of all molecules in the layer decrease as a result of increased surface coverage and the adsorption. The non-linear form of Temkin isotherm can be expressed by the following equation:

Table4.Non-linearisothermparametersfortheadsorptionofRB5ontoCMBPP.

Isotherm	Parameters	Values
Langmuir	<i>q_m</i> (mg/g)	211.8 ± 3.784
	K_L (L/mg)	0.0075 ± 0.0007
	R^2	0.9957
	χ^2	19.6
	RMSE	8.9
Freundlich	K_f (mg/g)	20.56 ± 1.466
	n	3.004 ± 0.405
	R^2	0.9676
	χ^2	145.8
	RMSE	69.4
Dubinin-Radushkevich	$q_m (mg/g)$	156.6 ± 9.16
	K	0.0654 ± 0.0136
	R^2	0.7573
	χ^2	559.5
	RMSE	176.3
Temkin	b (J/mol)	0.555 ± 0.04
	α (L/mg)	0.0812 ± 0.02
	R^2	0.9747
	γ^2	58.3
	RMSE	22.6



Figure 5. Non-linear isotherm modeling for Langmuir, Freundlich, Dubinin–Radushkevich and Temkin for the adsorption of RB5 onto CMBPP.

$$q_e = \beta \ln \alpha + \beta \ln C_e \tag{12}$$

where

 $\beta = \frac{RT}{b}$

In this study, the obtained value of b (0.555 ± 0.04 kJ/mol) was lower than 8 kJ/mol, showed that the heat of sorption of RB5 onto CMBPP is physical adsorption.

By comparing the correlation coefficient (R^2) values, it is observed that the Langmuir isotherm is the best model to describe the adsorption of RB5 onto CMBPP as it gave the highest R^2 of 0.9957. The adsorption parameters, along with the correlation coefficients (R^2), χ^2 and RMSE values of four non-linear isotherms are given in Table 4. Similarly, the error functions (χ^2 and RMSE) obtained for each isotherm model suggests the process can be explained in the order of Langmuir > Temkin > Freundlich > D–R. An example of this behavior is presented in Figure 5.

Comparison of CMBPP with other adsorbents

The sorption capacity of CMBPP obtained in the present work for adsorption of RB5 was compared with various adsorbent materials which have been reported in the literature (Table 5). From that comparison table, it is observed that CMBPP is an excellent adsorbent for RB5 than other adsorbents. Hence for the removal of RB5 from aqueous media, one can consider CMBPP as a potential candidate.

Effect of temperature

The adsorption of RB5 onto CMBPP carried out at various temperatures (298, 308, 318, and 303 K) at constant initial RB5 concentration of 300 mg/L (30 mL) and pH 3.0 is shown in Figure 3d. The experimental results showed that the adsorption (increased with rising in temperature) process is endothermic. To assure that the thermodynamic parameters (ΔG° , ΔH° , and ΔS°) for the sorption process (presented in Table 6) were calculated using the following equations:

$$\Delta G^{\circ} = -RT \ln K_c \tag{13}$$

Adsorbent	<i>q_{max}</i> (mg/g)	Optimum pH	References
Peanut hull	55.6	-	(Tanyildizi 2011)
Magnetic iron oxide nanoparticles	18	-	(Chang and Shih 2018)
Synthesized zeolite/bagasse fly ash	30	2.0	(Shah <i>et al</i> . 2011)
Aspergillus foetidus	92	3.0	(Patel and Suresh 2008)
Ionic liquid grafted-magnetic nanoparticles	161	5.5	(Poursaberi and Hassanisadi 2013)
Aqai stalks	153	4.0	(Cardoso <i>et al.</i> 2011)
Magnetic graphene oxide	188	3.0	(Kyzas <i>et al.</i> 2013)
Bone char	160	-	(lp et al. 2010)
Pumice	12.85	5.0	(Heibati <i>et al.</i> 2014)
AC from walnut wood	19.34	5.0	(Heibati <i>et al.</i> 2014)
CTS/MMT	82.64	2.0	(Umpuch and Sakaew 2015)
Treated dolomite (D900)	80.9	-	(Ziane <i>et al.</i> 2018)
Nano-ZnO/Chitosan composite beads	189.44	4.0	(Çınar <i>et al</i> . 2017)
CMBPP	211.8	3.0	In this study

$$K_c = \frac{C_{Ae}}{C_e} \tag{14}$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T \Delta S^{\circ} \tag{15}$$

$$\ln K_c = -\frac{\Delta G^{\circ}}{RT} = -\frac{\Delta H^{\circ}}{RT} + \frac{\Delta S^{\circ}}{R}$$
(16)

The slope (ΔH°) and the intercept (ΔS°) were calculated from the plot of $\ln K_c$ versus 1/T (Figure 6). The feasibility, spontaneity and more favorable at higher temperatures of the sorption process were understood by observing the decreased ΔG^o values from -0.0301 to -1.5535 with an increase in temperature. The obtained ΔG° , ΔH° values suggesting a physisorption process based on the following data. When the ΔG° value lies in the range of -20 to 0 kJ/mol it is physisorption, and -400 to -80 kJ/mol it is a chemisorption process (Munagapati et al. 2017). The positive value of ΔH° , indicating that the sorption process was endothermic. Additionally, the magnitude of ΔH° for absolute physisorption is <20 kJ/mol, and chemisorption is 80-200 kJ/mol. The positive value of ΔS° is interpreted as a sign of increment in randomness during adsorption of RB5 onto CMBPP at the interface.

Desorption and regeneration studies

To make the process more economical, it is essential to regenerate the biosorbent material, for that desorption studies were carried out. Similarly, this desorption study allows

 Table 6. Thermodynamic parameters for the adsorption of RB5 onto CMBPP at different temperatures.

Temp. (K)	ΔG^{o} (kJ/mol)	ΔH^{o} (kJ/mol)	ΔS^{o} (kJ/mol K)
298	-0.0301	-	-
308	-0.0482	15.02	0.051
318	-0.9688	-	-
328	-1.5535	-	-



Figure 6. Van't Hoff plot for the adsorption of RB5 onto CMBPP.

looking at mechanical aspects of the solute transfer process (Lakshmipathy and Sarada 2014). In the desorption studies deionized water, 0.1 M NaOH, 0.1 M CH₃COOH, 0.1 M HCl, 0.1 M H₂SO₄, 0.1 M HNO₃ were used as desorbing eluents. The percent desorption efficiencies with various desorbing eluents are shown in Figure 7a. From the results of this study, it has been observed that 0.1 M NaOH is the best desorbing eluent among the chosen ones for the recovery of CMBPP with 94% desorption.

In this study, regeneration experiments were carried out with 0.1 M NaOH for five adsorption-desorption cycles and the results were shown in Figure 7b. In the first cycle, the RB5 adsorption efficiency is found to be 88.5% that decreased to 78.6% in the fourth cycle and further decreased to 75.2% for the fifth cycle. But the CMBPP was quite stable even for the fifth cycle of regeneration study. Similarly, the desorption efficiency of 94.5% was observed in the first cycle that starts decreasing gradually and reached 82.3% after the fifth cycle of desorption. After that, it remains almost





Figure 7. Desorption studies of RB5: (a) desorption time with different desorbing agents, and (b) number of adsorption–desorption cycles with 0.1 M NaOH.

constant. These results confirmed that the CMBPP is an excellent reusable adsorbent and could be successfully utilized for the recovery of RB5 from water and wastewater.

Conclusions

This study investigated the adsorption of RB5 from aqueous solution on CMBPP used as adsorbent material and the result may be given as follows:

- The RB5 adsorption was dependent on the solution of pH, contact time, agitation speed, initial concentration of RB5 and temperature. The equilibrium adsorption reached within 180 min.
- The CMBPP was characterized by FTIR, SEM, BET and elemental analysis.
- The high pH-dependent adsorption behavior of CMBPP suggest the electrostatic attractions between CMBPP and RB5 play an essential role.
- The equilibrium adsorption data can be better explained (based on its R^2 , *RMSE*, and χ^2 values) by the non-linear models in the following order: Langmuir > Temkin > Freundlich > D-R.
- The Langmuir monolayer sorption capacity (q_{max}) of CMBPP was determined to be 211.8 mg/g at pH 3.0.
- The sorption capacity increased with increase in temperatures indicating the sorption process was endothermic.
- The pseudo-second-order better described adsorption kinetic data than the pseudo-first-order model.
- The thermodynamic parameters, such as ΔG° , ΔS° , and ΔH° was investigated. The negative and positive values of ΔG° and ΔH° showed that the adsorption of RB5 onto CMBPP is spontaneous and endothermic in nature.
- Regeneration studies exhibited that CMBPP could be effectively utilized for the recovery of RB5 and reused of adsorbent up to five adsorption-desorption cycles.

Overall, the results of this study showed that CMBPP is a suitable adsorbent for adsorption of RB5 from the aqueous solution.

Nomenclature

b	Temkin constant related to heat of adsorption, kJ/mol;
BET	Brunauer-Emmett-Teller;
C_{Ae}	amount of adsorbate adsorbed on the solid phase at equilib
	rium, mg/L;
C_e	remaining adsorbate in the liquid phase at equilibrium,
	mg/L;
C_i	initial RB5 concentration in the solution, mg/L;
FTIR	Fourier Transform Infrared Spectroscopy;
Κ	D-R isotherm constant;
k_1, k_2	pseudo-first-order (1/min) and pseudo-second-order (g/
	mg min) rate constants;
K _c	equilibrium adsorption distribution coefficient;
K_f	relative adsorption capacity, mg/g;
K_L	Langmuir isotherm constant, L/mg;
т	number of observations in the experimental design;
M	dry weight of CMBPP using in RB5 adsorption, g;
п	signifies the affinity of the adsorbate to the adsorbent;

p number of parameters;

q_e	quantity of RB5 adsorbed at equilibrium, mg/g;
$q_{e,m}$	equilibrium adsorption capacity obtained by calculating
	from the model, mg/g;
q_{e1}, q_{e2}	RB5 uptake at equilibrium, mg/g;
q_m	adsorption capacity of RB5 at equilibrium, mg/g;
q_t	uptake at any time <i>t</i> , mg/g;
Q	sorption capacity, mg/g;
R	ideal gas constant, 8.314 J/mol K;
SEM-EDX	Scanning electron microscopy with energy dispersive
	analysis of X-ray;
Т	absolute temperature, K;
V	volume of the solution, L;
ΔG^{o}	Gibbs free energy change, kJ/mol;
$\Delta H^{\rm o}$	enthalpy change, kJ/mol;
$\Delta S^{\rm o}$	entropy change, kJ/mol K;
ε	Polanyi potential;
α	equilibrium binding constant, L/mg.

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