



Potentiality of banana peel for removal of Congo red dye from aqueous solution: isotherm, kinetics and thermodynamics studies

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Received: 14 November 2017 / Accepted: 11 September 2018 / Published online: 18 September 2018
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Abstract

Potentiality of fruit peel (banana peel) was evaluated by adsorption of Congo red (CR) dye from aqueous solution. The adsorption study was performed by varying operating variables such as pH, initial concentration of CR, adsorbent dose, contact time and temperature. The dust of banana peel was characterized by different instrumental techniques such as point of zero charge, scanning electron microscopy and Fourier transformed infrared spectroscopy. The results revealed that the dye adsorption is favorable at higher pH (10). The equilibrium of dye adsorption was assessed by Freundlich and Langmuir isotherm models. Similarly, kinetics of CR adsorptions was assessed by pseudo-first-order and pseudo-second-order kinetics. Results revealed that isotherm and kinetics of dye adsorption fit well with Langmuir isotherm and pseudo-second-order kinetics, respectively. The dye adsorption capacity was recorded as 1.727 mg/g. The thermodynamics of the dye adsorption revealed that the adsorption is thermodynamically favorable, spontaneous and exothermic in nature. The regeneration study suggested that 97.3% dye removal can be achieved by regenerating the spent adsorbent with 0.1 N NaOH solution. Finally, it can be concluded that banana peel could be an efficient adsorbent for removal of Congo red from aqueous medium.

Keywords Adsorption · Banana peel · Congo red · Kinetics · Isotherms · Thermodynamics

Introduction

Dye industry is a major pollution generating industry including paper, painting, food processing, cosmetics, hair dye and textile (Yokwana et al. 2018; Munagapati and Kim 2016). It was estimated that more than 10,000 dyes are commercially available (Munagapati and Kim 2016). In the last 35 years, India has become a major producer of dyes and pigments in different industries such as textiles, paper, plastics, leather and cosmetics. Around 60% of the dyes used in the textile plants are azo dyes, which are characterized by azo ($-N=N-$) bond to the sp^2 hybrid carbon atoms (Fathi et al. 2015).

Congo red (CR) dye (1-naphthalenesulfonic acid, 3,3'-(4,4'-biphenylenebis(azo))-bis(4-amino) disodium salt) is a benzidine-based anionic diazo dye, which is known to metabolize the benzidine, a known human carcinogen (Shu et al. 2015). It was also reported (Munagapati and Kim 2016) that low concentration of CR dye affects the aquatic life and also leads to health hazardous symptoms such as difficulties in breathing, diarrhea, vomiting and nausea. There are numerous methods by which dyes can be removed from aqueous medium such as coagulation, flocculation, chemical oxidation, photochemical degradation and biological degradation. However, all the above-mentioned methods have some limitations, and none of them were successful in completely removing the color from wastewater (Sumanjit and Mahajan 2012). Adsorption is the most popular treatment process for the removal of dye from aqueous solution due to its simplicity, easy to recover and the reusability of the adsorbent.

Previous researchers highlighted varieties of biosorbents such as roots of *Eichhornia crassipes* (Wanyonyi et al. 2014), acid-activated red mud (Tor and Cengeloglu 2006), nanoparticle-coated activated carbon (Pal and Deb 2014),

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s13201-018-0811-x>) contains supplementary material, which is available to authorized users.

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chitosan (Li et al. 2015) and *Eichhornia crassipes* biomass (Roy and Mondal 2017), which have been extensively used for decontamination of CR dye from aqueous solution. However, none of the above-mentioned adsorbents showed excellent performance for Congo red removal. Therefore, some alternative adsorbents are necessary to fulfill the desired level.

In this present research, CR was selected as a dye to be removed from aqueous solution by banana peel dust. The adsorbent was characterized by FTIR and SEM studies. The influence of experimental parameters such as pH, adsorbent dose, contact time, initial dye concentration and temperature was investigated. Moreover, to investigate the adsorption process of CR using kinetics models such as pseudo-first order, pseudo-second order and intraparticle diffusion and thermodynamic studies. Finally, regeneration of the adsorbent was also performed.

Materials and methods

Materials and characterization

Adsorbent collection and preparation

The raw banana peels were collected from a local fruit market in Burdwan town. The materials were washed repeatedly with deionized water to remove soluble dust. The banana peels were allowed to dry in the sun for 5 h followed by hot air oven at 70 °C for 24 h (Fig. S1a and S1b). The dried fish scales were ground and sieved through metallic sieves ranging from 100 to 150 µm. The dried dust of banana peels was stored in a sterilized airtight plastic container.

Preparation of dye solution

A stock solution (1000 mg/L) of CR dye (Fig. S2) was prepared by dissolving 1 g CR dye in 1000 mL of double-distilled deionized water, which is the intermediate solution. The pH of the intermediate solution was adjusted by adding 0.1 M solution of NaOH and HCl using benchtop pH meter (EUTECH, pH 700). The concentration of dye solution was measured at 495 nm wavelength using PerkinElmer UV–Vis spectrophotometer (Perkin Lambda 35).

Characterization of the adsorbent

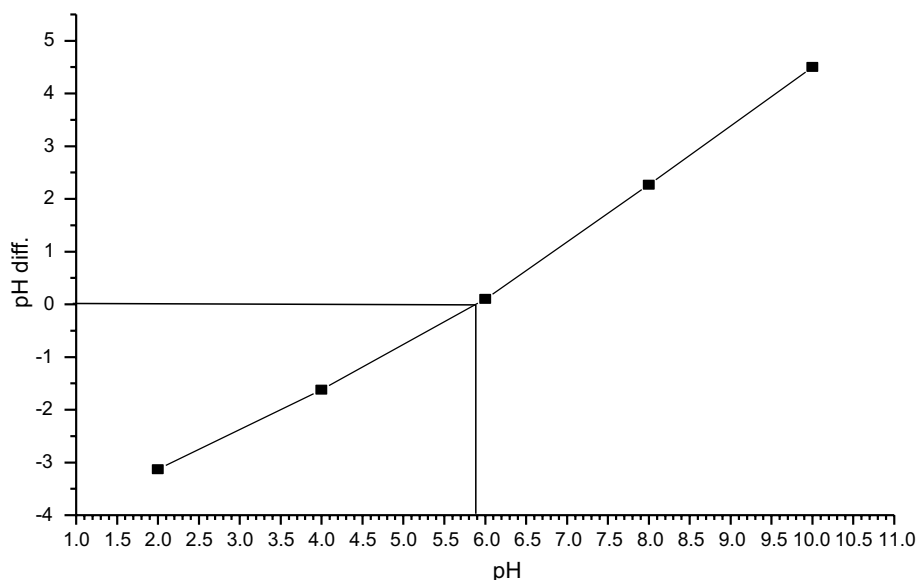
The point of zero charge of the adsorbent was determined by the solid addition method (Mondal and Roy 2015), and it is presented in Fig. 1. Surface morphology of the banana peel dust was studied by scanning electron microscopy (SEM) (HITACHI, S-530), and the surface chemistry was analyzed by Fourier transformed infrared spectroscopy (FTIR) (Bruker Tensor 27).

Batch adsorption study

In the batch adsorption study, the effect of different operating variables such as pH, initial concentration of dyes, adsorbent dose, contact time and temperature was considered for adsorption of CR. The amount of dye adsorption by fish scale dust at time t , q_t (mg/g) was calculated by the following mass balance relationship (Eq. 1):

$$q_t = \frac{(C_0 - C_t)}{m} \times V. \quad (1)$$

Fig. 1 pH_{ZPC} graph of banana peel adsorbent



The percentage of dye adsorption was calculated as (Eq. 2):

$$\% \text{Removal} = \frac{(C_0 - C_e)}{C_0} \times 100, \quad (2)$$

where C_0 is the initial dye concentration (mg/L), C_e is the equilibrium dye concentration at time t , V is the volume of solution (L) and m is the mass (g) of biosorbent.

Adsorbent regeneration study

The exhausted adsorbent was initially treated with 0.05 (M) NaOH solution followed by double-distilled water and finally dried in hot air oven at 60 °C for 24 h.

Results and discussion

Adsorbent characterization

Point zero charge

Alkali metal nitrate, especially potassium nitrate (0.01 N), is usually used to estimate the point of zero charge. The point of zero charge of banana peel dust (BPD) was determined, and the results are shown in Fig. 1. The pH_{pzc} values were known for determining the position where the resulting curves cut through the pH⁰ axis as shown in Fig. 1. For BPD, pH_{pzc} was recorded as 5.9. This value can be useful for predicting the surface charge of the adsorbents; when adsorption occurred at particular pH which is below the ZPC of the adsorbent, the surface of the adsorbent will be positive, and when adsorption occurred above the ZPC pH, adsorbent surface charge will be negative. The pH versus pH_{ZPC} of the banana peel dust is depicted in Fig. 1.

FTIR analysis

Fourier transform infrared spectroscopy is a valuable analytical tool by which the existence of important functional groups associated with the adsorbent can be identified (Mondal and Roy 2018). The origin and nature of the biosorbents, such as physical structure, chemical nature and functional groups, may control the biosorption performance. The FTIR spectra of banana peel before and after Congo red adsorption are presented in Fig. 2a and 2b, respectively. The spectra of both before and after adsorption of Congo red were taken in the range of 4000–500 cm⁻¹ to identify the functional groups involved in the biosorption. In both spectra, the broad and intense adsorption peaks in the range 3200–3500 cm⁻¹ reflected O–H stretching vibrations of hydroxyl group on the surface

of banana peel, corresponding to O–H stretching vibration of cellulose, pectin, adsorbed water, hemicelluloses and lignin. Before passing Congo red solution, the FTIR of banana peel showed that the peaks around 1639 cm⁻¹ are due to the C=C stretching that can be attributed to the presence of aromatic or benzene rings in lignin. An intense band in the area nearly 1450–1470 cm⁻¹ is supposed to the presence of (C–H) vibration of aliphatic and aromatic groups. The bonds in the range 1050–1450 cm⁻¹ can be assigned to the C–O stretching vibration of carboxylic acids and alcohols.

SEM study

There is an ever-increasing demand for the scanning electron micrograph in analytical field. The surface morphology of the adsorbent can be demonstrated by SEM photograph. In the present study, the micrograph of BPD before CR adsorption showed a heterogeneous, rough and porous nature. However, after adsorption of CR, the surface of BPD is almost smooth and homogeneous in nature (Fig. 3a, b). The similar structural observation of different adsorbents before and after adsorption of dye was reported by Zhang et al. (2011). Very recently, Sartape et al. (2017) highlighted in their study that *Limonia acidissima* (wood apple) shell showed rough surface before adsorption of malachite green dye. However, after dye adsorption, surface of *L. acidissima* shell is smoother in nature.

Effect of initial concentration

Figure 4 shows that the percentage of Congo red removal decreases with increasing initial concentration from 20 to 40 mg/L. That means higher level of dye adsorption occurred in lower concentration. Almost similar results of CR dye adsorption were experienced by Wang et al. (2014) in their work by using orange peel and rice husk as adsorbent. The adsorption capacity of the banana peel was also in accordance with initial concentration, except initial concentration 40 mg/L. However, opposite trend of q_e (mg/g) was recorded by Amel et al. (2012) for biosorption of cationic dye onto banana peel. They have recorded that adsorption capacity of cationic dye increases with increasing initial concentration of cationic dye. They also explained that higher concentration provides an important driving force to overcome all resistances of cationic dye between aqueous and solid phases, thus increasing the uptake. Moreover, they also established that increasing the initial concentration of cationic dye increases the number of collisions between the dye ions and the adsorbent, hence enhancing the adsorption process.

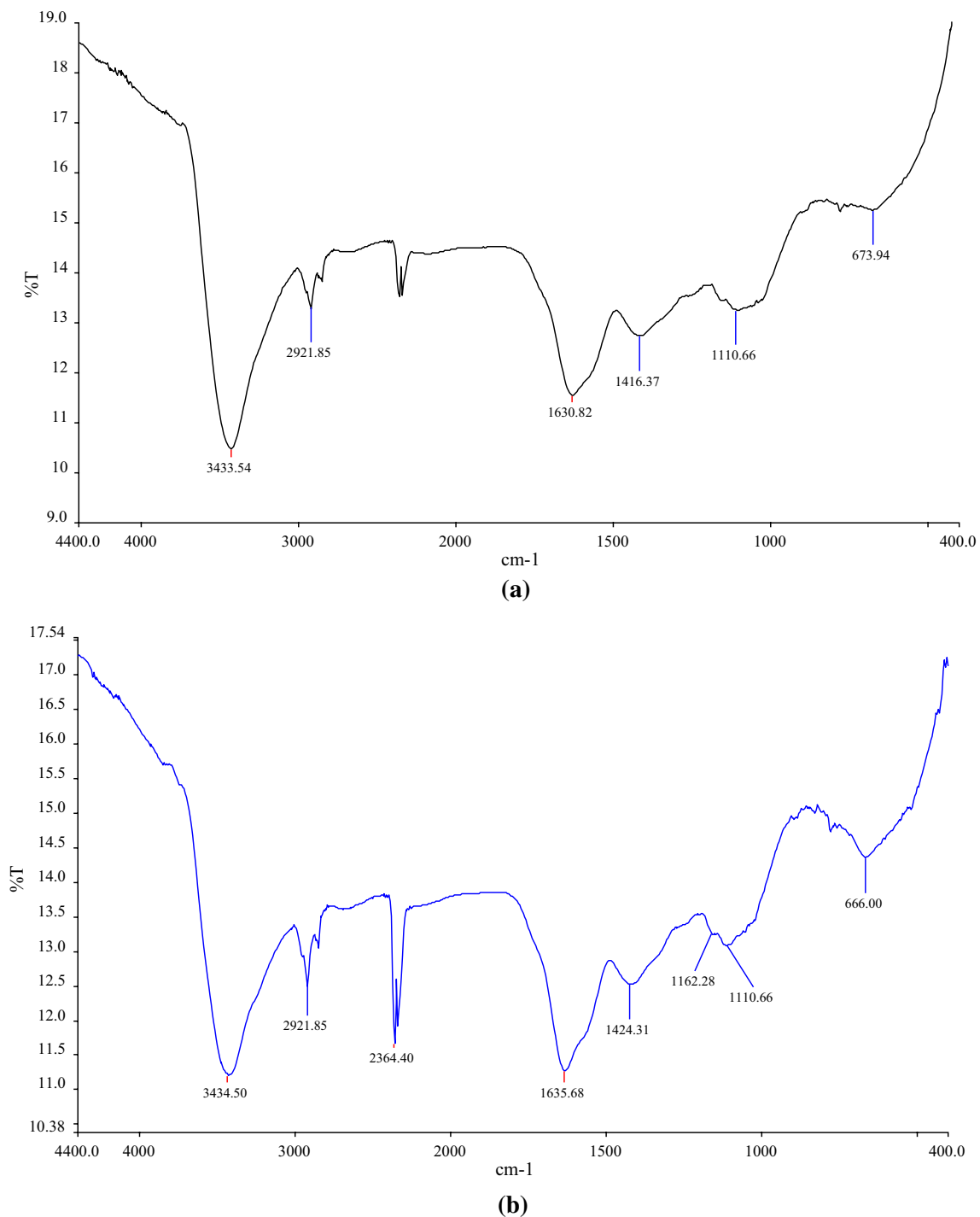


Fig. 2 FTIR spectra of BP **a** before and **b** after Congo red adsorption by banana peel

Effect of pH

The concentration of H⁺ or OH⁻ ion in the solution medium is the most vital for the adsorption of pollutants onto the adsorbents. In case of dye adsorption, the low and high pH is most influential than the medium pH (Priyantha et al.

2018). The final pH of an adsorption medium affects the adsorption mechanisms on the adsorbent surface and influences the nature of the physicochemical interactions of the species in solution and the adsorptive sites of adsorbents. In the present experiment, the effect of pH was identified by adding 80 mL of Congo red dye with an agitation speed

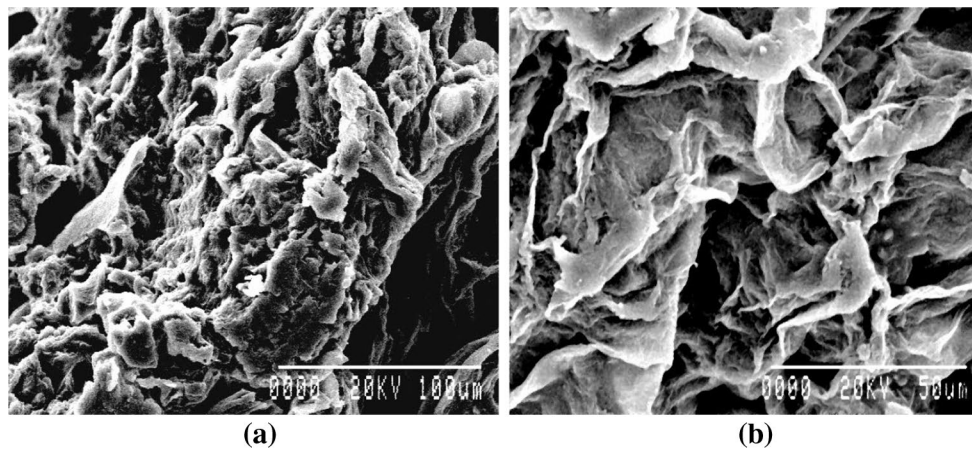
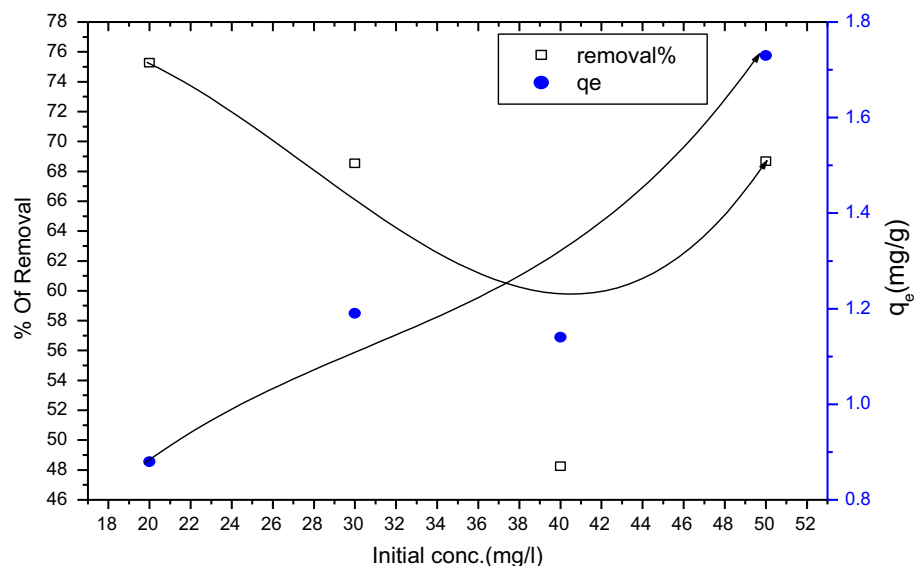


Fig. 3 **a** Banana peel before exposed with Congo red dye. **b** Banana peel after exposed with Congo red dye

Fig. 4 Effect of initial concentration of CR dye on adsorption banana peel



of 600 rpm for 90 min at a constant temperature 40 °C. The effect of pH on the adsorption of Congo red dye by banana peel at pH ranging between 3 and 12 is shown in Fig. 5. The adsorption efficiency increased from 57% (0.61 mg/80 mL) to 75% (0.88 mg/80 mL) when the solution pH varied from 3 to 12. About 8% improvement in removal efficiency was recorded above neutrality. However, maximum removal was recorded at pH 10. This is probably due to maximum ionization of dyes under alkaline pH. BPD material is composed of various functional groups, such as amino and carboxylic, which could also be affected by the pH (Achak et al. 2009). Moreover, BPD also consists of various biomolecules such as protein, carbohydrate, oxalate, phytate and saponin, which are also strongly influenced by pH of the medium (Achak et al. 2009; Anhwange et al. 2009). The high removal efficiency at higher pH (10) is probably due to hydrogen bonding or weak force between adsorbent and adsorbate. As the

pH_{ZPC} value is 5.9, the pH above 5.9, the adsorbent surface gets negative charge and dye is adsorbed completely through hydrogen bonding. A similar observation was reported for the adsorption of CR onto different adsorbents (Saha et al. 2013; Torkian et al. 2012).

Effect of adsorbent dose

The amount of adsorbent is the most valuable parameter. This is because the active surface of the adsorbents solely depends on its amount (Sartape et al. 2017). Adsorbent dose is an important parameter because it determines the capacity of an adsorbent for a given initial concentration of the adsorbate. The equilibrium uptake for the adsorption of CR onto banana peel was recorded as 0.88 mg/80 mL. The system was agitated for 90 min (600 rpm) at a constant temperature 40 °C at the initial pH 10. Figure 6

Fig. 5 Effect of pH on the adsorption of CR dye by the banana peel

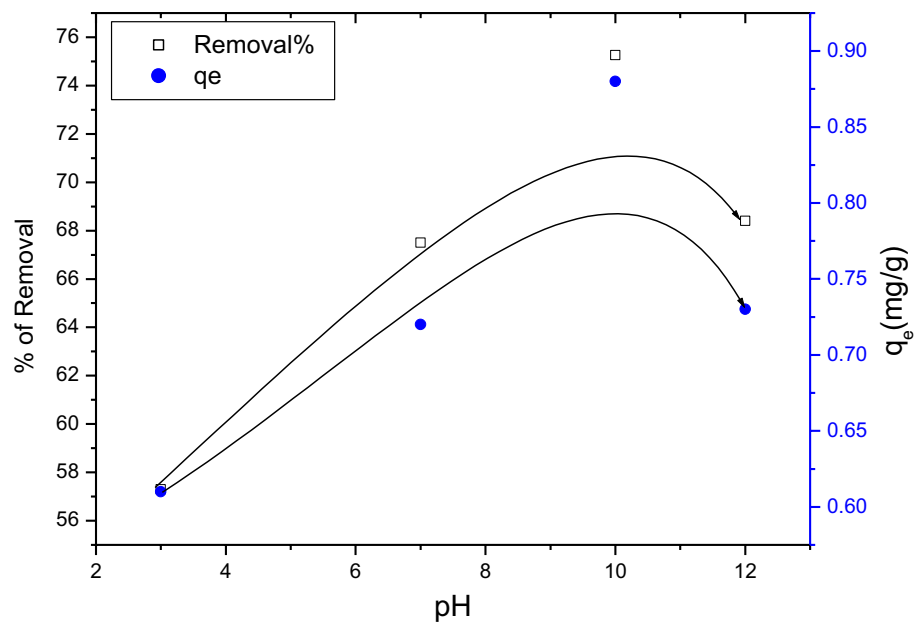
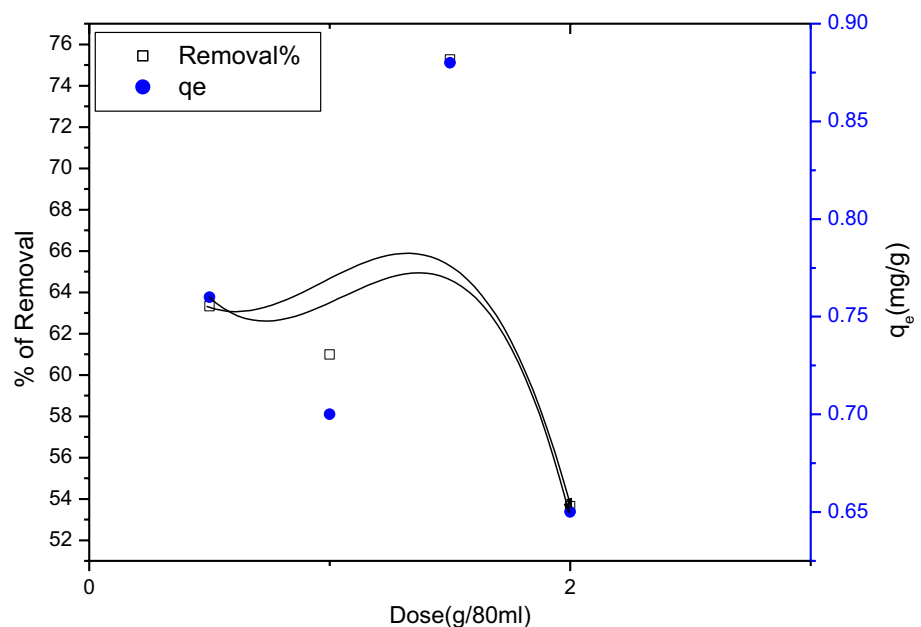


Fig. 6 Effect of adsorbent dose on the adsorption of CR dye by banana peel



shows that the increase in adsorbent from 0.5 g/80 to 1.5 g/80 mL resulted in the percentage of removal from 63.32 to 75.26%. It is readily understood that the number of available adsorption sites increases with the increase in the adsorbent dose and it, therefore, results in the increase in the amount of adsorbed CR dye. The increase in adsorbent rate over 1.5 g/80 mL has not allowed any additional improvement in adsorption. This seems to be due to the binding of almost molecules of CR to the sorbent and establishment of equilibrium between the molecules bounded to the sorbent and unadsorbed molecules in the

solution. Similar observation was recorded by Achak et al. (2009) for removal of phenolic compounds by low-cost biosorbent 'banana peel.' The adsorption capacity was initially high, for low adsorbent dose. However, many factors can contribute to this adsorbent concentration effect. The most important factor is the adsorption sites that remain unsaturated in the adsorption reaction. The decrease in adsorption capacity with an increase in adsorbent dose is mainly attributed to the fact that at higher dose, active sites of the adsorbents were reduced due to aggregation (Garg et al. 2004; Han et al. 2008).

Effect of contact time

Effect of contact time on adsorption of CR dye by banana peel is presented in Fig. 7. The adsorption of CR was continued up to 90 min which corresponds to 75% of CR adsorption. After reaching maximum adsorption at 90 min, no further improvement was recorded. Moreover, the results also revealed that the adsorption capacity was recorded very fast in the first 30 min. This is perhaps due to the initial availability of maximum number of active sites which gets saturated with time. As a result, the remaining vacant surface sites

are difficult to be occupied due to the formation of repulsive force between the Congo red compounds on the solid surface and the bulk phase (Srivastava et al. 2006).

Effect of stirring rate

The effect of stirring rate on CR adsorption was investigated at 40 °C with 20 mg/L and 1.5 g/80 mL banana peel. The experiments were carried out by agitation speed at a stirring rate of 100 to 600 rpm. As shown in Fig. 8, 75.40% of CR was obtained at 200 rpm, dropped to 70.82% at

Fig. 7 Effect of stirring time on the adsorption of CR dye by banana peel

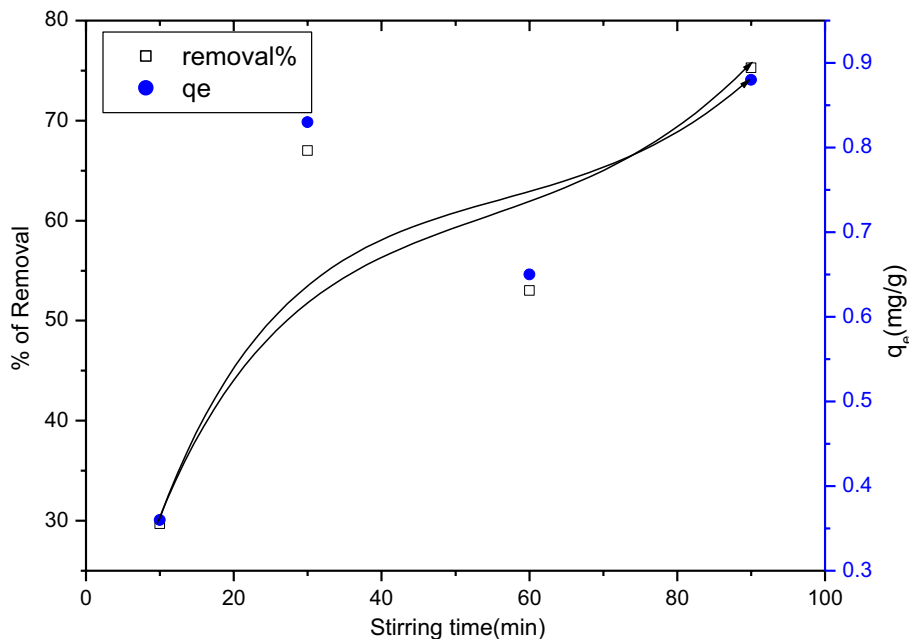
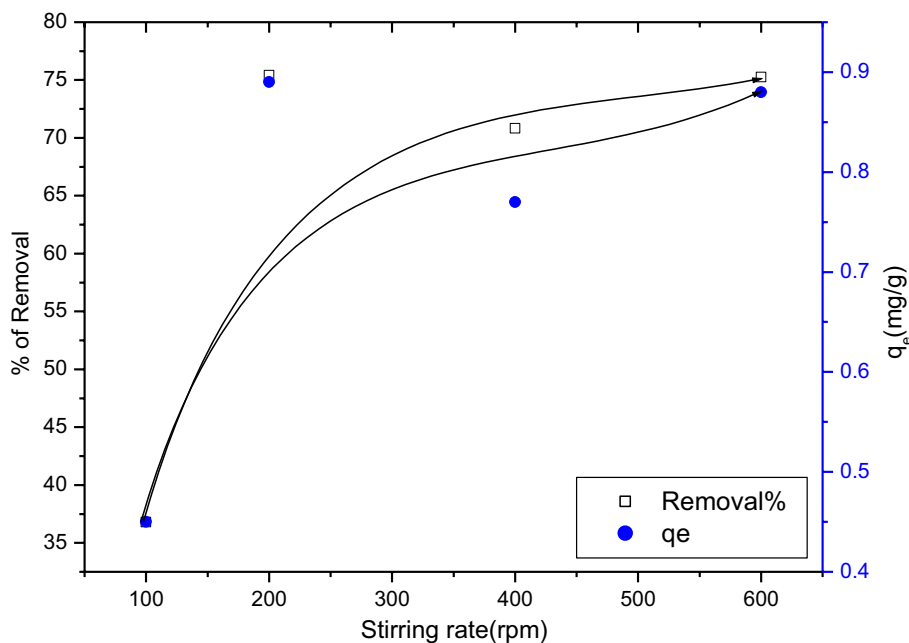


Fig. 8 Effect of stirring rate on the adsorption of CR dye by banana peel



400 rpm. Initially there is a steady increase in CR removal with increasing stirring rate and reached maximum (75.40%) at 200 rpm. The percentage of removal after 200 rpm is probably due to the decrease in attractive force between the lignocellulosic functional groups (having carboxylic and alcoholic groups) and CR molecule.

Effect of temperature

The temperature is the most crucial factor, which directly influences the adsorbent–adsorbate interaction (Bhaumik et al. 2017). It was experienced that at higher temperature the adsorbate molecules diffuses out from the external boundary as well as opens up the internal pores which subsequently reduced viscosity of the solution (Dogan et al. 2004). Increasing the temperature is known to increase the rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the adsorbent particles as results of the reduced viscosity of the solution (Dogan et al. 2004). In addition, changing the temperature alters the equilibrium capacity of the adsorbent for a particular adsorbate (Dogan et al. 2004). The thermodynamic parameters such as enthalpy and entropy change can be understood with the variation of temperature change in the adsorption process (Alkan et al. 2004). Figure 9 illustrates the effect of temperature on the adsorption of CR initially at 50 mg/L onto banana peel as a function of time. The equilibrium adsorption capacity was clearly affected by temperature in the adsorption by CR. Adsorption increased from 56% ($q_e = 0.598$) to 75.40% ($q_e = 0.89$) when the temperature was raised from 32 to 60 °C. This 19% increase in CR removal with 8% rise of temperature indicates not

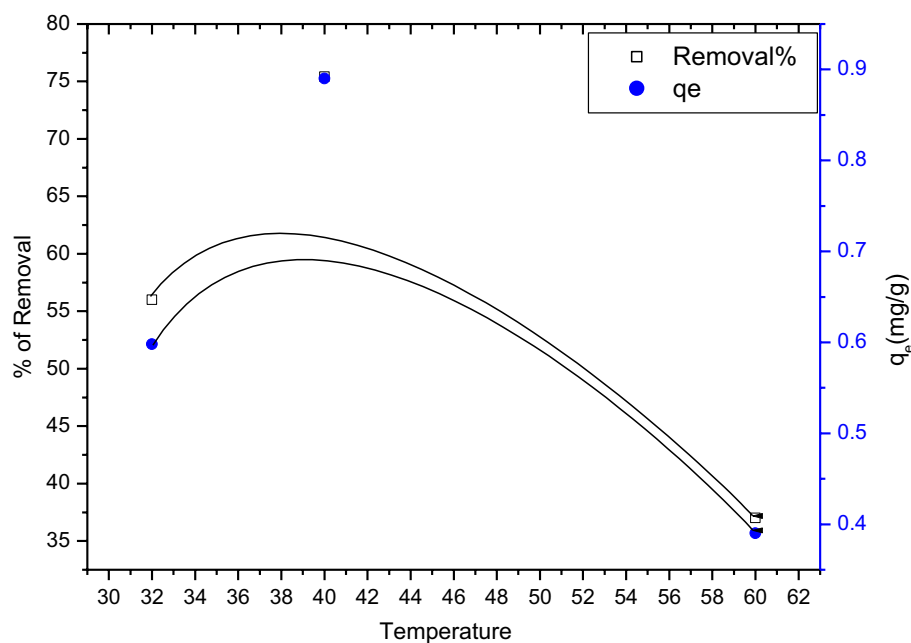
only that temperature has a significant effect on adsorption (Gundogan et al. 2004) but also that the adsorption of CR onto the banana peel adsorbent surface is an endothermic process (Almeida et al. 2009). Before equilibrium was reached, increased temperature led to an increase in the rate of CR adsorption, implying a kinetically controlling process, as found in many other systems (Ozcan et al. 2006). The mobility of molecules increases generally with a rise in temperature, thereby facilitating the formation of surface monolayers (Ozcan et al. 2006). The increase in CR adsorption with rise in temperature clearly indicates that this particular dye adsorption is endothermic in nature (Munagapati and Kim 2016). Very recently, Banerjee and Chattopadhyay (2017) reported that dye uptake capacity of sawdust was increased with increasing temperature from 298 to 318 K, and they suggested that the diffusion of dye from the surface of adsorbent is controlled by a diffusion mechanism. Previous research also suggested that at higher temperature pore size of the adsorbent increased.

Adsorption isotherms

Adsorption properties and equilibrium data, known as adsorption isotherms, describe how pollutants interact with adsorbent materials and are very important for optimization of adsorption system. Equilibrium relationship between sorbent and sorbate is described by sorption isotherms, usually the ratio between the quantity adsorbed and that remaining in the solution at a fixed temperature at equilibrium (Turabik 2008; Crini 2006).

There are several isotherm models available for analyzing experimental data and for describing the equilibrium of

Fig. 9 Effect of temperature on the adsorption of CR dye by BP



adsorption. The equilibrium isotherm for the adsorption of Congo red dye on adsorbent was determined. The q , K_L , K_F , n and R^2 (correlation coefficient) are given in Table 1. It is clear from Table 1 that the CR adsorption data are fit well with Langmuir isotherm. This result basically indicates the monolayer adsorption on the surface of homogeneous binding sites of the adsorbent (Roy et al. 2014).

Adsorption kinetics

Adsorption mechanism can be demonstrated with the help of several models. In order to investigate the mechanism of sorption, characteristic constants of sorption were determined using a pseudo-first-order equation of the Lagergren based on solid capacity and a pseudo-second-order equation based on solid-phase sorption (Nandi et al. 2009a, b; Barka et al. 2011). The kinetic parameters and coefficient of determination (R^2) for each model are presented in Table 2. Moreover, the theoretical q^2 values calculated by the pseudo-second-order model were close to the experimental q_e values. On the basis of these results, it can be concluded that the pseudo-second-order kinetics model provides a good correlation for the adsorption of CR by banana peel dust.

Adsorption thermodynamics

Consideration of energy is an important parameter for the determination of spontaneity of process in adsorption chemistry. Thermodynamic parameter values are the indicators of practical application of a process.

The van't Hoff equation is as follows (Eq. 3):

$$\ln K_0 = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}, \tag{3}$$

where K_0 is the rate of the equilibrium concentration of the dye ions on adsorbent to the equilibrium concentration of dye ions in solution. R is the ideal gas constant (8.314 J/mol K) and T is the adsorption temperature in Kelvin. The chemical potential being referred to is the Gibbs free energy

Table 1 Isotherm model parameters for the adsorption of CR onto banana peel

Isotherm	Parameters	Values
Langmuir	q_m (mg/g)	1.721
	K_L (L/mg)	0.204
	R^2	0.988
	χ^2	23.28
	Freundlich	K_f (mg/g)
	N	3.745
	R^2	0.877
	χ^2	64.73

Table 2 Kinetics parameters for the adsorption of CR onto banana peel

Kinetic model	Parameters	Values
Pseudo-first order	q_e , exp (mg/g)	0.853
	q_e , cal (mg/g)	0.622
	k_1 (L/min)	0.0092
	R^2	0.766
	χ^2	54.06
Pseudo-second order	q_e , cal (mg/g)	0.749
	k_1 (L/min)	0.237
	R^2	0.918
	χ^2	16.34

(ΔG kJ/mol) of the component in individual phases. Free energy can be calculated using enthalpy (ΔH kJ/mol) and entropy (ΔS kJ/mol).

A plot of $\ln K_0$ versus $1/T$ should give a straight line from where (ΔH°) and (ΔS°) can be obtained and ΔG° calculated. Table 3 shows the results of the thermodynamic parameters. The positive ΔH° value reveals that the adsorption process is endothermic (Vimonses et al. 2009). The negative values of ΔG° obtained at 32° and 40 °C indicate that the adsorption of CR on BP is spontaneous in nature (Hu et al. 2010) and the shift of ΔG° value from -0.625 to 1.473 with increasing the temperature is indicative of a slow and less spontaneous adsorption process at the higher temperature.

Dye desorption study

Desorption study is an important step for the regeneration of exhausted adsorbent and recovery of the adhering pollutants (Mondal et al. 2015). However, recovery of the adsorbent required proper selection of the eluents, which strongly depends on the type of adsorbent and the adsorption mechanism (Munagapati and Kim 2016). The present study clearly suggests that among the eluents 0.1 N HCl, 0.1 N NaOH, 0.1 N CH_3COOH and distilled water, maximum percentage of recovery of CR was 97.3% with 0.1 N NaOH (Fig. 10). Almost similar regeneration of

Table 3 Thermodynamic parameters for the adsorption of CR onto banana peel

Dye	Thermodynamic parameters				
	T (°C)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (kJ/mol)	R^2
Congo red			-28.29	-0.087	0.395
	32	-0.625			
	40	-2.919			
	60	1.473			

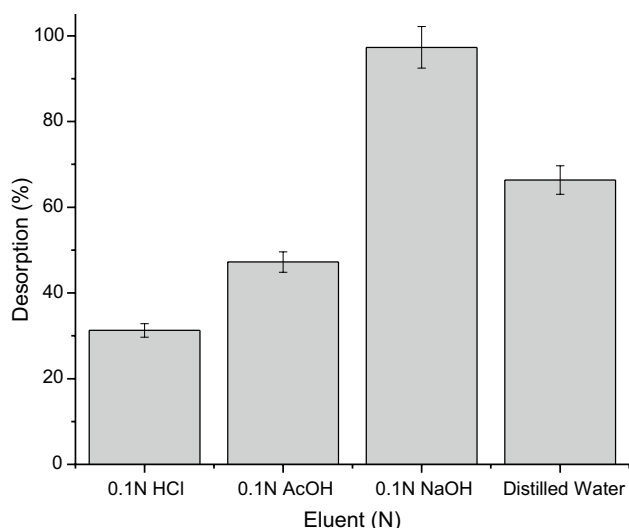


Fig. 10 Desorption of Congo red by different eluents

Table 4 Comparative study of Congo red adsorption performance

Adsorbent	Capacity (mg/g)	References
<i>Saccharum bengalense</i>	125	Din et al. (2013)
Pineapple plant stem	11.966	Chan and Ong (2016)
Xanthan gum/silica hybrid nanocomposite	209.205	Ghorai and Pal (2013)
Root of <i>Eichhornia crassipes</i>	90%	Wanyonyi et al. (2014)
Raw pine cone powder	32.65	Dawood and Sen (2012)
Zinc peroxide nanomaterial	208	Chawla and Singh (2017)
Banana peel powder	1.721	Present study

cationic modified orange peel powder saturated with CR was reported by Munagapati and Kim (2016).

Comparative study with previous findings

The performance of dye removal by banana peel dust was compared with the previous study, and the results are presented in Table 4. From Table 4, it is clear that xanthan gum/silica hybrid nanocomposite, *Saccharum bengalense* nanomaterial, showed an excellent performance for the adsorption of Congo red. However, pineapple plant stem, root *Eichhornia crassipes* and raw pine cone powder are exhibited moderate performance for the adsorption of Congo red. But the present adsorbent, banana peel dust, showed much lower adsorption capacity of Congo red (1.721 mg/g).

Conclusion

This study demonstrated that banana peel is suitable adsorbent for CR removal in a batch study. The interactions between CR and functional groups on the banana peel surface of the adsorbent were confirmed by FTIR analysis. Moreover, SEM study revealed that the surface heterogeneity of the banana peel was reduced after adsorption of CR. The various operating parameters such as initial concentration of CR, pH, adsorbent dose, contact time, shaking rate (rpm) and temperature were found to be effective in the adsorption process. The adsorption on isotherm fit with Langmuir adsorption isotherm, and monolayer adsorption capacity of CR was recorded 1.721 mg g⁻¹. The kinetic studies revealed that the adsorption process nicely fit with pseudo-second-order kinetic model. Thermodynamic studies confirmed that the process was endothermic and spontaneous up to 40 °C. Finally, exhausted adsorbent was regenerated by treating with different effluents. Therefore, based on the results, it can be concluded that banana peel is an effective and efficient adsorbent for the removal of CR from aqueous solution.

Acknowledgements The authors acknowledge their sincere thanks to the funding agency, DST Government of India F. No. SR/FST/ESI-141/2015(C), dated May 26, 2016, for providing necessary funds for conducting the present research. The authors are also thankful to all faculty members and nonteaching staff of the Department of Environmental Science, University of Burdwan, West Bengal, India, for providing infrastructural facilities and active moral support for the completion of this work.

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