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Research Article

KINETIC AND ISOTHERMAL STUDIES ON THE REMOVAL OF BRILLIANT BLUE FCF FROM AQUEOUS SOLUTION USING WALNUT SHELL POWDER

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ABSTRACT

In the present study, walnut shell powder (NIWSP) was explored as potential adsorbent for removal of Brilliant Blue FCF (BBF); a toxic triphenylmethane dye from aqueous solution using batch process. The parameters, such as, initial dye concentration, adsorbent dosage, temperature and pH, influencing the process of biosorption were studied. Kinetic studies were performed using pseudo first-order and pseudo second-order equations. The absorption parameters were determined using Langmuir and Freundlich isotherm models. The latter gave good fitting to the experimental data. The thermodynamic parameters, like, change in enthalpy (ΔH°), entropy (ΔS°) and Gibbs free energy (ΔG°) of adsorption system were also determined and evaluated. Scanning electron microscopy and Fourier transform infrared spectroscopy were used for the surface characterization of the adsorbent.

INTRODUCTION

Synthetic dyes are extensively used in industries (Srivastava, Sinha & Roy 2004) with the main purpose of enhancing aesthetic appeal. The textile industry has a major role in the economy. It is estimated that about 700,000 tons of dyes and pigments are produced annually (Zollinger 2003; Robinson *et al*., 2001, Ogugbue & Sawidis 2011) and a major portion of these dyes enter the environment through wastewater. The complex molecular structure of synthetic dyes makes them difficult to get biodegraded, and as such they are the principle pollutants in aquatic environment (Forgacs, Cserhati & Oros 2004; Przystaś, Zabłocka-Godlewska & Grabińska-Sota 2012). The hydrolysed dye Reactive Blue 19 as for example has about 46 years of half-life at pH 7 and 25°C (Hao, O. J., Kim, H., & Chiang, P. C. 2000; Firmino *et al*., 2010). Thus, removal of dyes from waste water before their disposal is extremely important (Ogugbue & Sawidis 2011, O'Neill *et al*., 1999).

Carneiro *et al*. (2010) developed and optimized, accurate and sensitive analytical method for monitoring dyes like C.I. Disperse Blue 373 (DB373), C.I. Disperse Orange 37 (DO37) and C.I. Disperse Violet 93 (DV93) in environmental samples. Their result showed the presence of DB373, DO37 and DV93 in both drinking water and untreated river water, thereby proving that the effluent treatments like pre-chlorination, coagulation flocculation and, flotation generally used by drinking water treatment plants, were not effective in removing these dyes. It was also confirmed by the detection of mutagenic activity in these wastewaters (Carneiro *et al*., 2010). Hence the development of any effective new method(s) for the removal of dye is the need of the present day.

Brilliant Blue FCF (BBF) is a triarylmethane dye having reddish-blue powder. It is extensively used as a colouring agent for foods (wiki/Brilliant_Blue_FCF). It is suspected that it can induce allergic reactions in individuals with pre-existing asthma (wiki/Brilliant_Blue_FCF). In 2003, the U.S. FDA issued a public health advisory to warn health care providers of the potential toxicity of this synthetic dye in enteral feeding solutions (wiki/Brilliant_Blue_FCF). The following legal limits apply in the EU (E 131) and other countries: 150–300 mg/kg depending on the type of food; safety limit for foods and drugs: 0.1 mg/day per kg body weight (wiki/Brilliant_Blue_FCF). The ADI for Brilliant Blue FCF is 6 mg/kg. It is soluble in water, and has λ_{max} of 628 nm. Currently, the process of biosorption, which is defined as the removal of materials (organic compounds, metal ions, dye molecules, etc.) by inactive, non-living biomass (materials of biological origin) is an eco-friendly technology as against the existing costly water treatment technologies due to its low initial cost, simplicity of

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design, ease of operation, insensitivity towards toxic substances, and almost complete removal of pollutants even from dilute solutions (Saha *et al.*, 2012).

Low-cost sorbents derived from various agricultural waste, industrial waste and activated carbon and multiwall carbon nano tubes have been investigated for removal of dyes from aqueous solutions (Bhatnagar, A., & Jain, A. K. 2005; Kumar, Miranda &Velan 2005; Rajappa, Ramesh & Nandhakumar 2017). To handle the large volume of effluents generated by food industry, spent/waste generated by nutraceutical industries which are available in large quantities could be appropriately used for the purpose.

Juglansregia (walnut tree) is native to the region stretching from the eastward Balkans to the Himalayas and southwest China (wiki/Juglansregia). Different parts of walnut tree have a long history of medicinal use, being used in folk medicine to treat a wide range of diseases (Miliauskas, Venskutonis, & Van Beek 2004). Different parts of Walnut tree are being used as Nutraceutical. A nutracerutical is defined as, "a food or a part of a food that provides medicinal or health benefits, including the prevention or treatment of a disease" (FBrower 1998).The word Nutraceutical was used firstly by Stephen DeFelice MD, founder and Chairman of the foundation for innovation in medicine (FIM) (Brower 1998). The nutraceuticals differ from dietary supplements in that the nutrceuticals are not only supplement the diet but also aid in the prevention or treatment of disease. Nutraceuticals are also represented as conventional food or as the sole item of meal or diet (Kalra 2003). The leaves are alterative, anti-inflammatory, anthelmintic, astringent and depurative. They are used internally in the treatment of chronic cough, constipation, diarrhoea, dyspepsia, and asthma (www.naturalmedicinalherbs.net).The benefits of walnut are attributed to its rich and various nutritional components. It is considered among the nuts with the highest caloric content (about 642 kcal/100g) (foodsanddiseases.com). About 14.3% of proteins, lowest in carbohydrates (only 13.5%), rich in B group vitamins, mainly vitamin B1 (which is important for the functioning of heart) and vitamin B6 (foodsanddiseases.com). Walnuts are rich in potassium, phosphorus, and trace elements like copper, zinc, and manganese (foodsanddiseases.com). Medicinal applications of walnuts include useful lowering of cholesterol level, effective in preventing or treating coronary heart disease, for the diabetics due to its very low carbohydrate content, for neurons metabolism, it is recommended invariably for those suffering from depression, stress and irritability(foodsanddiseases.com). Walnuts are grown in India in Himachal Pradesh, Arunachal Pradesh, Jammu Kashmir and Uttrakhand. Jammu and Kashmir state produces around 98% of the country's output [28]. It is projected that the total requirement for walnut in India will increase from the present demand of 36,000 tonnes to 72,550 tonnes by 2020 (www.actahort.org).Walnut consists of two parts namely, fruit and the outer shell. The outer shell is a hard material extensively used as fuel which increases carbon foot print. The weight of the fruit vis-à-vis outer shell is about 60 %,which amounts to thousands of tons which at present are used as a fuel. Thus, walnut shell, a waste material is explored as a biosorbent for the remediation of BBF dye. Various factors affecting adsorption capacity like initial dye concentration, adsorbent dosage, temperature, and pH, has been evaluated. In order to optimize the conditions for maximum adsorption different models of isotherms and adsorption kinetics are fitted to the experimental data to establish the removal capacity of this adsorbent. The Fourier transform infrared spectroscopy (FTIR) used for characterisation of NIWSP and scanning electron microscopy (SEM) analyses were used to assess the changes in adsorbent material before and after dye adsorption.

MATERIAL AND METHODS

Materials

Brilliant Blue FCF, disodium; 2-[[4-[ethyl-[(3-sulfonatophenyl) methyl] amino] phenyl]-[4-[ethyl-[(3-sulfonatophenyl) methyl] azaniumylidene] cyclohexa-2, 5-dien-1ylidene] methyl] benzenesulfonate is a triarylmethane dye. Its chemical formula is$C_{37}H_{34}N_2Na_2O_9S_3$ and molecular weight 792.85. It has absorbance maximum (λ_{max}) 628nm and classified as C.I. 42090. It was procured from Sigma Aldrich Pvt Ltd, Mumbai, India. The chemicals used throughout the study were of analytical-grade reagents. A stock dye solution of 1000 mg/L was prepared by dissolving known amount of dye in distilled water. All working solutions of required concentrations were prepared by diluting the stock solution with distilled water for later use. The molecular structure of BBF is shown in Fig. 1.

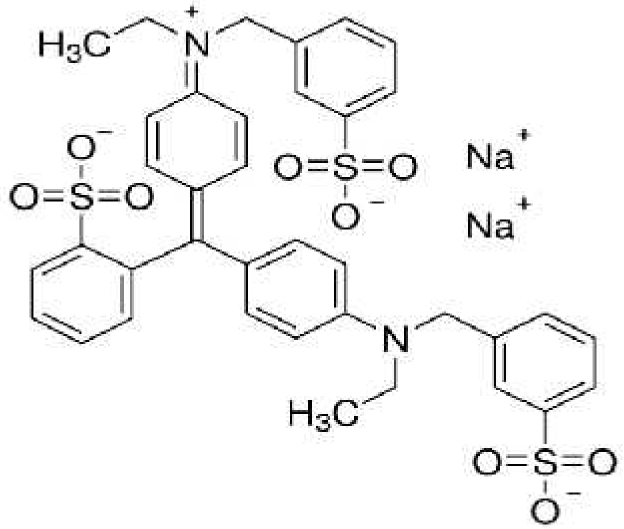


Fig. 1 Chemical Structure of Brilliant Blue FCF

Preparation of adsorbent

The Walnut shell was procured from a local industry. The NIWSP was dried at 60°C for 24 h in an oven. The dried NIWSP was ground and passed through ASTM sieve 80 mesh to get the particle size of ≤ 177µm, and stored in plastic jar for later use. Prior to adsorption process no other chemical or physical treatments were given to NIWSP.

Surface characterization

NIWSP was observed through Scanning Electronic Microscope (Zeiss Evo/LS15, Germany). The functional groups present in the adsorbent were identified by FTIR spectrometer (Inter-spec 2020, Spectro Lab, UK).

Adsorption

Adsorption of BBF from aqueous solution by NIWSP was investigated by batch method. The effects of various parameters affecting adsorption, such as, initial dye concentration, adsorbent dosage, temperature and pH were studied.

Batch adsorption experiments were carried out by adding a fixed amount 50 mg of NIWSP into 250mL Erlenmeyer flasks containing 50mL of initial dye concentration (10-100 mg/L). The flasks were agitated (Kemi Orbital Shaker, India) at 170 rpm at 28±2°C for 180 min, until equilibrium was reached. The samples were centrifuged at 3000 rpm for 10 min. The supernatant liquid containing un-adsorbed dye solution was removed carefully using micropipette and the absorbance of the colored solution was measured by a double beam UV/Vis spectrophotometer (Perkin Elmer- Lambda 25, USA) at 628 nm. The amount of BBF adsorbed at equilibrium, q_e(mg/g) was calculated using following equation (1).

$$q_e = \quad C_0 - C_e \frac{V}{W} \qquad \ldots\ldots(1)$$

Where, C_0 and C_e are concentrations (mg/L) of BBF at initial and at equilibrium respectively, V is solution volume (L) and W is adsorbent weight (g).For kinetic studies, the same procedure was followed. The concentrations of BBF were similarly measured. The amount of BBF adsorbed at any time, q_t (mg/g), was calculated using equation (2).

$$q_t = \quad C_0 - C_t \frac{V}{W} \qquad \ldots\ldots(2)$$

Where, C_t (mg/L) is the concentration of BBF measured at time t. Initial concentrations of 25, 50 and 100 mg/L of the dye and adsorption time of 15 min (2 min intervals) were used. For determining optimum amount of adsorbent per unit mass of adsorbate, 50 mL of dye solution was mechanically stirred in an orbital shaker with different amounts of NIWSP (0.025-0.200 g/L) till equilibrium was attained. To study the influence of pH on dye adsorption, 50mg of NIWSP along with 50mL of dye solution of concentration 200mg/L were agitated using orbital shaker. The experiment was done with pH values of 2-12.The pH was adjusted with dil HCl and/or NaOH solution. Solution pH was determined by pH meter (Systronics 802, India). Agitation was continued for 180 min. It is observed that 140-150 min and with constant agitation speed of 170 rpm is sufficient to reach the equilibrium. However, agitation speed of 170 rpm for 180 min was fixed for all studies. At equilibrium, the dye concentration was measured using double beam UV/Vis spectrophotometer at 628 nm. The extent of removal of dye was determined by following equation (3).

$$Dye\ removal\ efficiency\,\% = \frac{C_0 - C_e}{C_0} \times 100 \ldots\ldots(3)$$

Where, C_o and C_e are concentrations (mg/L) of BBF at initial and at equilibrium.

Modeling studies

Adsorption isotherms

Adsorption isotherm models provide information about interaction mechanisms, surface properties and affinities of adsorbent. The most accepted models for single solute system with two parameters are Langmuir and Freundlich. The models were used to test the equilibrium adsorption at ambient temperature.

The non-linear forms of isotherm models studied are shown below.

Freundlich isotherm: $\ln q_e = \ln K_F + (1/n) \ln C_e$ (4)

Langmuir isotherm: $C_e / q_e = (1/bq_{max}) + (1/q_{max}) C_e$ (5)

Where, q_e is the amount of dye at equilibrium in unit mass of adsorbent (mg/g), C_e is concentration of dye solution at equilibrium (mg/L), q_{max} and b are the Langmuir coefficient related to adsorption capacity (mg/g) and adsorption energy (L/mg), respectively. K_F and n are the Freundlich coefficient related to adsorption capacity [(mg/g) / (mg/L) $^{1/n}$] and adsorption intensity of adsorbent, respectively. In our present work only Freundlich model fits to the data.

Adsorption kinetics

The controlling of the adsorption process was done by fitting experimental data with pseudo- first-order and pseudo-second-order. The controlling mechanism of the adsorption process was found by fitting the experimental data with the respective kinetic equations.

Thermodynamic parameters

Energy and entropy enable to understand the feasibility and mechanism. In the present study, thermodynamic parameters, including standard free energy (ΔG^0), enthalpy change (ΔH^0) and entropy change (ΔS^0) were estimated by using rate law and also kinetic data to find out the extent and enthalpy of the adsorption process.

$$\log (q_e m/C_e) = \Delta S^\circ / 2.303R + (-\Delta H^\circ/2.303RT) \qquad (6)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \qquad (7)$$

Where, m is the adsorbent dose (g/L), C_e is concentration of dye solution at equilibrium (mg/L), q_e is the amount of dye adsorbed at equilibrium in unit mass of adsorbent (mg/g), q_e/C_e is the adsorption affinity. ΔH°, ΔS° and ΔG° are change in enthalpy (kJ/mol), entropy (J/mol/K) and free energy (kJ/mol), respectively, R is the gas constant (8.314 J/mol/K) and T is the temperature (K).The values for ΔH° and ΔS° were obtained from the slopes and intercepts respectively. From the Van't Hoff plots of log ($q_e m/C_e$) *versus* 1/T, ΔG^0 values were obtained from Equation 7.

RESULTS AND DISCUSSION

Surfacecharacterization

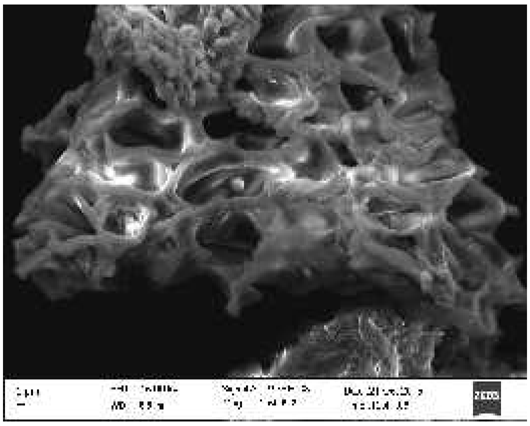
Scanning Electron Microscopy

The NIWSP morphology obtained by SEM exhibited fibrous and porous structure as shown in Fig. 2(A). This structure enhancesdye adsorption as observed in Fig. 2 (B) where the pores and voids between the spaces areoccupied by the dye.

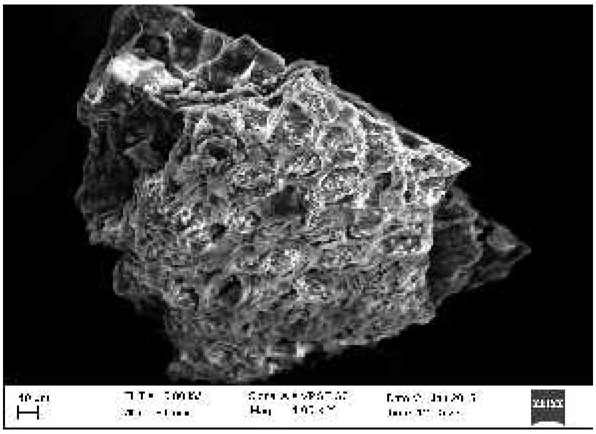
FTIR characterization of NIWSP

As seen in Fig.3, The FTIR spectroscopy of NIWSP showed the broad band around 3178 cm^{-1} is attributed to the surface hydroxyl groups, linked in cellulose and adsorbed water, B and at 1622.06cm^{-1} is due to C=C stretching of olefins. The band at

1406.36 cm^{-1} indicates methyl groups. The band at 1082.69 cm^{-1} is due to the presence of cellulose in adsorbent.



A



B

Fig 2 SEM Image of NIWSP before A and after B adsorption

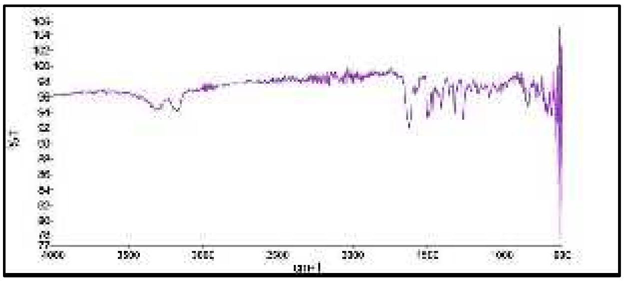


Fig 3 FTIR of NIWSP before adsorption

Adsorption of BBF

Effect of initial dye concentration

As seen in Fig 4, the dye uptake increased from 6 to 51 mg/g of NIWSP with the increase in dye concentration from 10 to 100 mg/L respectively. This indicates that there is an increase on the driving force of the concentration gradient due to the increase in the initial dye concentration. Adsorption was rapid initially due to the dye getting adsorbed onto exterior surface. Later, the dye molecules, probably entered into pores (interior surface), which is relatively as low process. The adsorption of BBF was more with higher concentration and remained almost constant after equilibrium time.

Influence of adsorbent dosage

The influence of adsorption of the dye onto NIWSP increased with the enhancement in the adsorbent dosage from 0.025 to 0.200 g. This may be due to the binding of almost all dye molecules on the adsorbent surface and establishing equilibrium of dye molecules and the adsorbent. Hence dye adsorption increased with adsorbent dosage and reach equilibrium at certain dosage the results are shown in Fig.5.

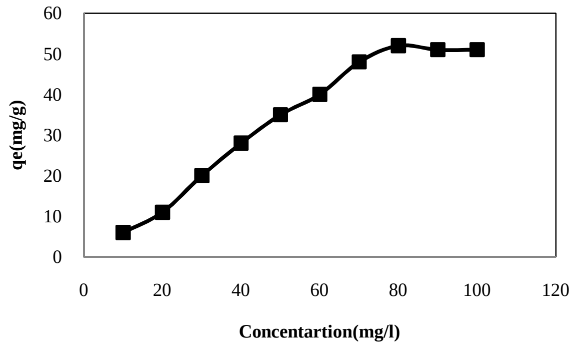


Fig.4 Effect of initial dye concentration on adsorption of BBF

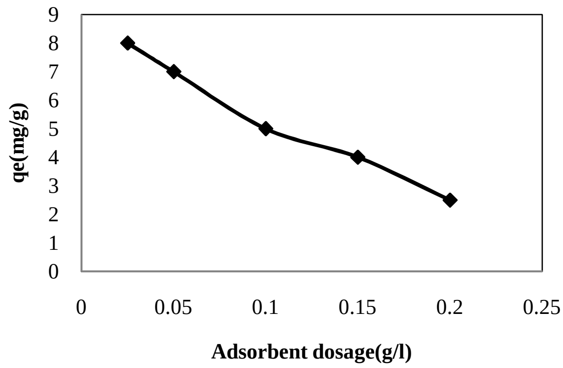


Fig 5 Effect of adsorbent dosage on adsorption of BBF

Effect of Temperature

Temperature is an influencing factor in the adsorption process and it was studied at 30°C, 40°and 50°C and the results are shown in Fig.6. It can be observed that with the increase in temperature, the adsorption capacity increases marginally, which indicates that the process is exothermic in nature.

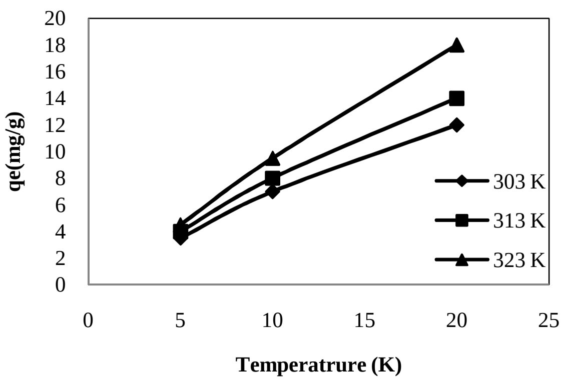


Fig 6 Effect of Temperature on adsorptionof BBF

The increase in adsorption with temperature may be due to the increase in the mobility of the dye molecule with increase in their kinetic energy and the enhanced rate of intra-particle diffusion of adsorbate with the rise in temperature. The slight increase in removal of dye due to increasing temperature may be due to higher interaction between adsorbate and adsorbent.

Effect of pH

pH is one of the most important parameters in the adsorption process. It controls the adsorption capacity by influencing adsorbent surface properties and ionic forms of dye. The adsorption capacity of NIWSP slightly increased with increase in solution pH and maximum adsorption capacity of BBF was under acidic condition. In acidic pH an excess of H^+ ions compete with cations of the dye for adsorption sites. This result in increased adsorption and increase in pH decreases the adsorption results are depicted in Fig. 7.

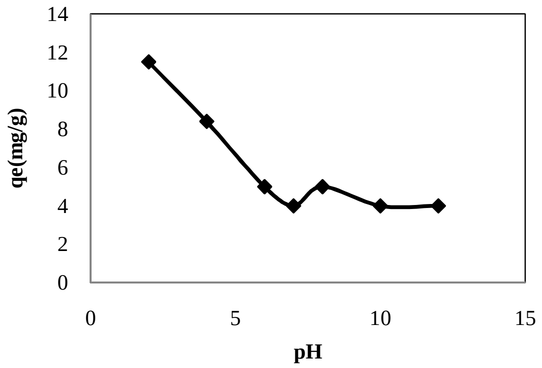


Fig 7 Effect of pH on adsorption at 20 ppm concentration of BBF

Adsorption isotherms

The equilibrium experiments were conducted for different initial concentrations of BBF in the range of 10-100 mg/L. The adsorption of the dye onto adsorbent using Langmuir Model assumes the monolayer adsorption on to the surface of adsorbent containing finite number of identical adsorption sites of uniform energies (Langmuir 1917). The Langmuir isotherm parameters q_{max} and b were determined from slope ($1/q_{max}$) and intercept ($1/bq_{max}$) of the plot of C_e /q_e*versus*C_e(Fig. 8).

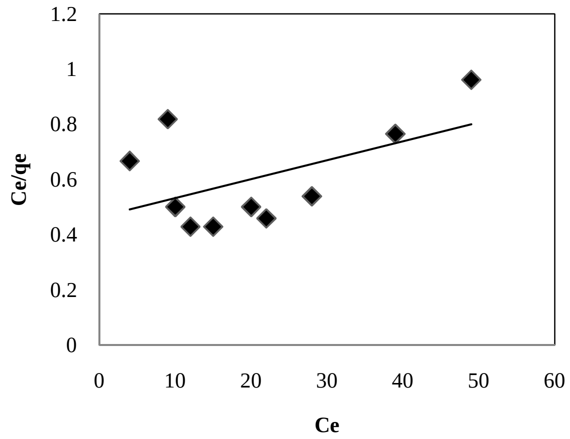


Fig 8 Fitting of experimental data to Langmuir model

The values of Freundlich constants n and K_F were obtained from the slope (1/n) and intercept (lnK_F) of the plot of lnq_e *versus* lnC_e (Fig. 9) respectively. The plot gives a straight line and this shows that adsorption of BBF on NIWSP also follows the Freundlich isotherm. Freundlich isotherm model (Freundlich 1906) is an empirical equation which assumes that the adsorption process takes place on heterogeneous surface. The heterogeneity factor (n_F) indicates whether the nature of adsorption is linear (n_F=1), chemisorption (n_F < 1), or physisorption (n_F > 1). In the present study, the value of n_F is 1.096 indicates that the adsorption is physisorption and favours normal Langmuir isotherm. The fitting of Langmuir and Freundlich isotherms to the experimental data R^2 is 0.27 and 0.85 respectively; where, R^2 is the correlation coefficient shows that the data fits well. The results are presented in Figs. 8 and 9. Hence it is inferred that the adsorption of BBF on NIWSP is favourable and the process is physisorption. The Langmuir and Freundlich isotherms constants and regression coefficients are listed in Table 1.

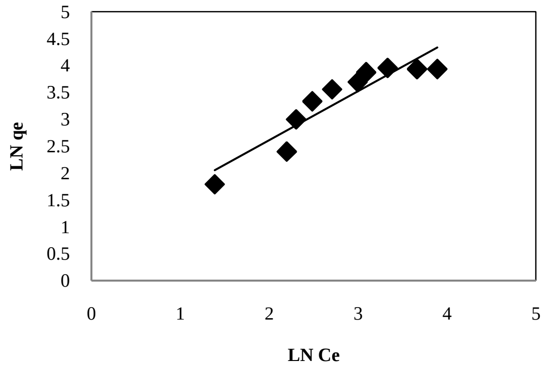


Fig 9 Fitting of experimental data to Freundlich model

Table 1 Isotherm parameters for BBF adsorption on NIWSP

Freundlich Constants			Langmuir Constants		
K_F	n_F	R^2	q_{max}	b	R^2
2.195	1.096	0.855	147.05	0.0068	0.275

Adsorption kinetics

The rate constants for the adsorption of BBF on NIWSP were obtained using the pseudo-first-order and pseudo-second-order kinetic models.

Pseudo-first-order kinetic model

When the adsorption is preceded by diffusion through a boundary, the kinetics in most cases follows the pseudo-first-order rate equation. The differential rate equation is as follows

$$dq_t /dt = k_1 (q_e - q_t) \quad (8)$$

Where, q_t and q_e are the amounts of dye adsorbed at time t (mg/g) and at equilibrium (mg/g), respectively and k_1 is the pseudo-first-order rate constant (min^{-1}). Integration of the above equation by using the boundary condition, q_t = 0 at t = 0 gives equation 9 (Largegren 1898):

$$\log (q_e - q_t) = \log q_e - (k_1/2.303)\, t \quad (9)$$

The values of k_1 and q_e were calculated from the slopes and intercepts of the linear plots of log (q_e − q_t) *versus* t, and presented in Table 2. There is a great disagreement between experimental and calculated values of q_e as shown in Table 2. Therefore, it may be concluded that the adsorption of BBF onto NIWSP has not followed the pseudo-first-order kinetic model.

Pseudo-second order kinetic model

The pseudo-second-order kinetic model (Ho& McKay 1998) is presented in equation 10:

$$dq_t /dt = k_2 (q_e - q_t)^2 \quad (10)$$

Where, q_t and q_e are the amount of dye adsorbed at time t (mg/g) and at equilibrium (mg/g), respectively and k_2 is the pseudo-second-order rate constant (g/mgmin). Integrating the above equation using the boundary condition, q_t= 0 at t = 0 leads to equation 11:

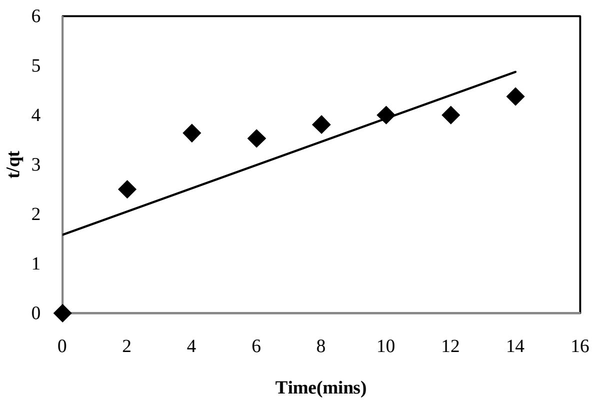


Fig 10 Pseudo-Second order kinetic model for 5ppm of BBF

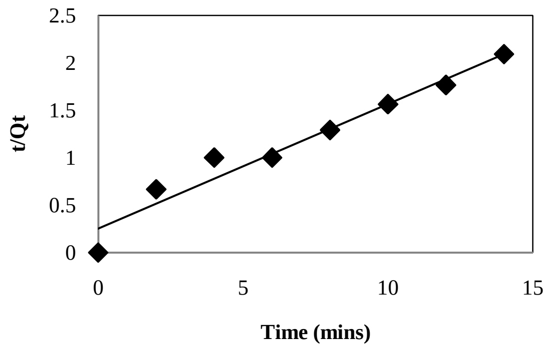


Fig. 11 Pseudo-Second-order kinetic model for 10ppm of BBF

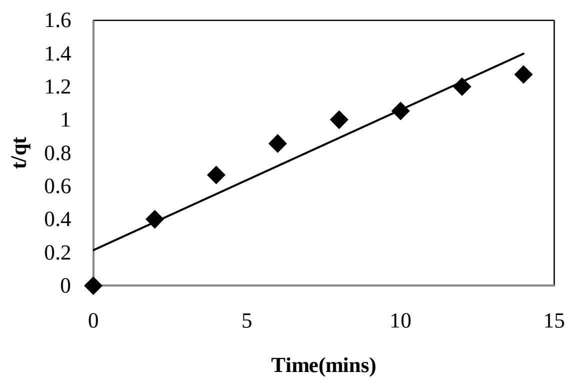


Fig 12 Pseudo-Second-order kinetic model for 20ppm of BBF

$$t/q_t = 1/k_2 q_e^2 + t/ q_e \quad (11)$$

The values of k_2 and q_e were calculated from intercepts and slopes of the linear plots of t/q_t *versus* t respectively and presented in Table 2. Table 2 shows that the calculated q_e values are very close to that of experimentally obtained q_e and the values of correlation coefficients (R^2) are closer to unity confirms that adsorption of BBF onto NIWSP follows pseudo-second-order kinetics.

Table 2 Kinetic parameters for BBF adsorption on NIWSP

Concentration of Dye (ppm)	Pseudo first order			Pseudo second order		
	q_e (mg /g)	K_1(min^{-1})	R^2	q_e (mg /g)	K_2(min^{-1})	R^2
5	1.852	0.166	0.931	4.253	2.682	0.660
10	2.184	0.244	0.941	7.604	30.284	0.953
20	2.760	0.145	0.947	11.820	55.209	0.918

Effect of Adsorption thermodynamics

The Gibbs free energy, entropy and enthalpy changes of adsorption were calculated by Van't Hoff and Gibbs-Helmholtz equations. As seen in Table 3, the positive ΔH° value suggests the endothermic nature of adsorption while the low magnitude and very similar values of enthalpy irrespective of changes in initial dye concentration also clearly indicate that the adsorption is physical i.e., involving weak interactions. The ΔG° is negative for all studied temperatures indicating that the adsorption of BBF onto NIWSP follows a spontaneous and favourable trend. The decrease in ΔG° with increase in temperature indicates increase in adsorption at higher temperatures. The positive value of ΔS° suggests good affinity of BBF towards the adsorbent and increased randomness at the solid solution surface.

Table 3 Thermodynamic parameters for BBF adsorption on NIWSP

Concentration (mg/L)	ΔH^0	ΔS^0	$-\Delta G^0$		
	(kJ/mol)	(J/mol/K)	(kJ/mol)		
			303 K	313 K	323 K
5	54.3	199.3	6.01	8.00	9.99
10	83.8	295.6	5.73	8.49	11.63
20	71.6	251.8	4.66	7.15	9.67

CONCLUSIONS

The major conclusions of this study are:

- NIWSP has been developed as efficient, environmental-friendly and cost-effective biosorbent for the remediation of dye Brilliant Blue FCF.
- Operational parameters such as; initial dye concentration, adsorbent dose, temperature and pH, influenced the adsorption efficiency of NIWSP.
- The experiments and analyses of the results for adsorption isotherms and kinetic studies confirmed the complexity involved in the dye adsorption mechanism.
- Thermodynamic study demonstrated the spontaneous and endothermic nature of biosorption process. It also confirmed that the adsorption is physical in nature.
- An alternative use of walnut shell as a biosorbent will help to reduce carbon foot print.

- NIWSP is appropriate ready-to-use matrix in field of adsorption science.
- The authors envisage that the concept demonstrated will help overcoming resource depletion through utilization of agro-based spent which has neither feed or fertilizer value.

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