

Sequestration of Brilliant Green Dye by Coriander Leaf: Isotherm and Kinetic Studies

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Abstract

Adsorption process has been performed to investigate the potentiality of coriander leaf to sequester brilliant green (BG) dye from the aqueous phase. Coriander leaf of 75-micron size has been characterized by SEM, BET, FTIR etc. In the present study, emphasis has been given on the removal of BG dye under different conditions *viz.* dose of biomaterial (10.0 - 100.0 mg), pH (2.0 - 12.0) and dye concentration (10.0 - 100.0 mg/L) by Taguchi optimization. The batch experiments have been conducted at 25 °C with stirring speed of 120.0 rpm. The kinetic study is performed at two different temperatures, 30 and 40 °C. The batch adsorption process for sequestration of the dye followed pseudo-2nd order kinetics ($R^2 = 0.99$). The study also reveals that the Freundlich isotherm is the best fitted ($R^2 = 0.96$) model. The adsorption capacity is 68.49 mg/g and the overall removal efficiency is above 80.0 %.

Keywords: Adsorption, Coriander, Pseudo-2nd order, Isotherm, Taguchi optimization

Introduction

Sequestration of dyes and pigments from the polluted water is an objective of immense importance in a diversified environmental field. Dyes and their intermediates (7×10^5 ton per annum) used in various industries generate large amount of wastewater, and thereby causes environmental pollution [1-3]. Brilliant green (BG) belongs to triarylmethane dyes and widely used in various sectors *viz.* textile, pharmaceutical, ink and paint industries etc. [4,5]. In spite of its lot of applications, it causes irritation to the gastrointestinal tract, nausea, vomiting and diarrhea in human depending on the exposure time and concentration [6]. Therefore, the sequestration of such coloring agent is necessary.

Several treatment methods *viz.* biological treatment, coagulation, nano-filtration, ozonation, photo degradation and adsorption are often applied for the removal of dyes [7]. Among processes used to sequester dyes from the aqueous phase, adsorption using biomass, is much more convenient due to high efficiency, easy availability, low-cost, renewable and less sensitivity towards toxic substances [8]. Thus, considerable attention should be placed to explore different types of waste biomass as suitable adsorbents for removal of dyes and pigments in view of environmental sustainability as well as economical feasibility.

Coriander (*Coriander sativum*) is an annual herb and cultivated in almost all countries throughout the year. Seeds are used in food flavoring industries (bakery and confectionary) and leaves are used as ingredient in food dishes. Coriander (leaf, root and seed) has also some medicinal values due to presence of polyphenols, coriandrol, monoterpene hydrocarbons, heterocyclic compounds, isocoumarin, phthalides, phenolic acids, sterols etc. [9]. Leaves contain vitamin A, B₂ (riboflavin), C and dietary fiber. Nadeem *et al.* [10] analysed the protein, fat, starch, vitamin, mineral contents of fresh leaves of coriander.

It is revealed from the literature that several research works have been carried out on coriander seeds and its waste for the effective removal of heavy metals (Cu^{+2} , Zn^{+2} , Pb^{+2} , Hg^{+2} , CH_3Hg^+) [11,12] and dyes [13]. Nutraceutical industrial coriander seed spent has adsorption potentiality for removing of acid black 52, congo red, methylene blue, ethidium bromide and acid blue 113 [13 and references therein]. Few research works have been conducted to understand the potentiality of coriander leaf to remove environmental pollutants. The above objective has been realised in the present work via the use of easily available, low-cost and non-toxic coriander leaf as an adsorbent for the effective removal of Brilliant green dye under the influence of different parameters along with Taguchi optimization. Isotherm, kinetics and thermodynamic studies have also been performed to explore the insight of adsorption

behaviour. This study might be helpful for the valorisation of biomass derived from the coriander as an adsorbent for the remediation of dye contaminated effluent.

Materials and methods

Biomass and dye

Coriander leaves have been collected from the local market of Tribeni, West Bengal. It is washed with tap water, dilute acid and base (0.1 (N) HCl and 0.1 (N) NaOH), distilled water to obtain the neutral pH. The leaves are dried at 105 °C for 24 h, ground and sieved (sieve size 75 micron), named CL-75, and kept in the air tight container. The BG is purchased from Himedia Pvt Ltd.

Instruments and software

The adsorbent (CL-75) is characterized by the FTIR with the help of Shimadzu FTIR 8400 spectrophotometer. The BET analysis is performed using the nitrogen adsorption-desorption isotherm at degassing temperature of 120 °C. The absorption spectra have been measured by Shimadzu UV-1700 spectrophotometer. Orion 4-star Benchtop pH meter and Orbital shaker of AB-SB systems India have also been used. The physicochemical properties, point of zero charge (pZC) and zeta potential have also been studied following standard procedure [14]. Statistical analyses have been performed by MINITAB 14 software.

Adsorption experiment

The potentiality of CL-75 as bio-adsorbent has been investigated by selected variable parameters viz. time, dose, concentration and pH. The study has been conducted with the aqueous solution of BG (50.0 mL) with varied concentrations (10.0, 20.0, 30.0, 40.0, 50.0, 60.0, 70.0, 80.0, 90.0 and 100.0 mg/L) and CL-75 (50.0 mg) in the batch mode in an orbital shaker (120.0 rpm at 25 °C). Kinetic studies have been performed at 30 and 40 °C, and the different pH values are adjusted by adding 0.1 (N) NaOH/HCl solutions. The concentrations of BG dye are measured from absorbance values at wave length 627.0 nm. The rate of adsorption (R %) of the dye onto CL-75 is calculated by Eq. (1), and the equilibrium adsorption quantity of CL-75 for dye, q_e (mg/g), is calculated by Eq. (2).

$$R(\%) = \frac{C_0 - C_e}{C_0} \times 100 \% \quad (1)$$

$$q_e = (C_0 - C_e) \times \frac{V}{W} \quad (2)$$

where C_0 and C_e represent initial and equilibrium concentrations of the dye respectively (mg/L in the liquid-phase, V is the volume of the experimental solution (l), and W is the weight (g) of CL-75 used.

Results and discussion

Characterization of the adsorbent

The adsorbent material (CL-75) is characterized by FTIR, BET surface area, zeta potential, point of zero charge and physico-chemical properties have been estimated (**Table 1**). The particle charge demand is negative which means that CL-75 may adsorb positively charged molecules. The pH_{PZC} of CL-75 is 6.45 implies that below pH_{PZC} ($pH < pH_{PZC}$) value, the number of positively charged sites of adsorbent (CL-75) increase and favours anionic dyes to adsorb on the surface. On the other hand, above pH_{PZC} ($pH > pH_{PZC}$) value, the number of negatively charged sites of adsorbent (CL-75) increase and favours cationic dyes to adsorb. The measured values of BET surface area (S_{BET}), pore volume and pore radius further facilitate the adsorption. The BET surface area (S_{BET}) is low (16.003 m²/g) and compared with reported values of other bio-adsorbents [15].

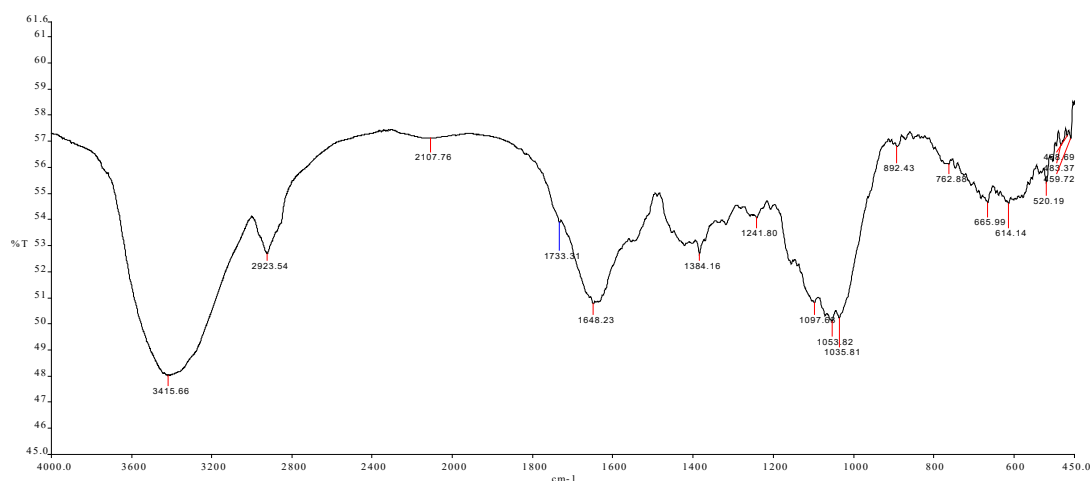
The FTIR spectra of the adsorbent (**Figures 1(a) and 1(b)**), CL-75 have been analysed before and after the dye adsorption. The characteristic peaks at 3400, 2925, 2110, 1648, 1384, 1241, 1097, 1053, 1035, 892, 762, 665 and 614 cm⁻¹ correspond to various functional groups, such as alcoholic –OH or amine –NH, alkyl –CH stretching, alkyne –C≡C, aromatic –C=C and aromatic –CH stretching, mainly –OH and –NH groups are responsible for binding of BG. Appearance of a new peak at 1544 cm⁻¹ due to N–N stretching frequency may indicate the adsorption of BG on CL-75 and compared with the reported data [16].

The SEM morphologies of CL-75, before and after adsorption are shown in **Figures 2(a) and 2(b)** respectively. The micrograph of CL-75 exhibits highly porous, irregular, heterogeneous structure with

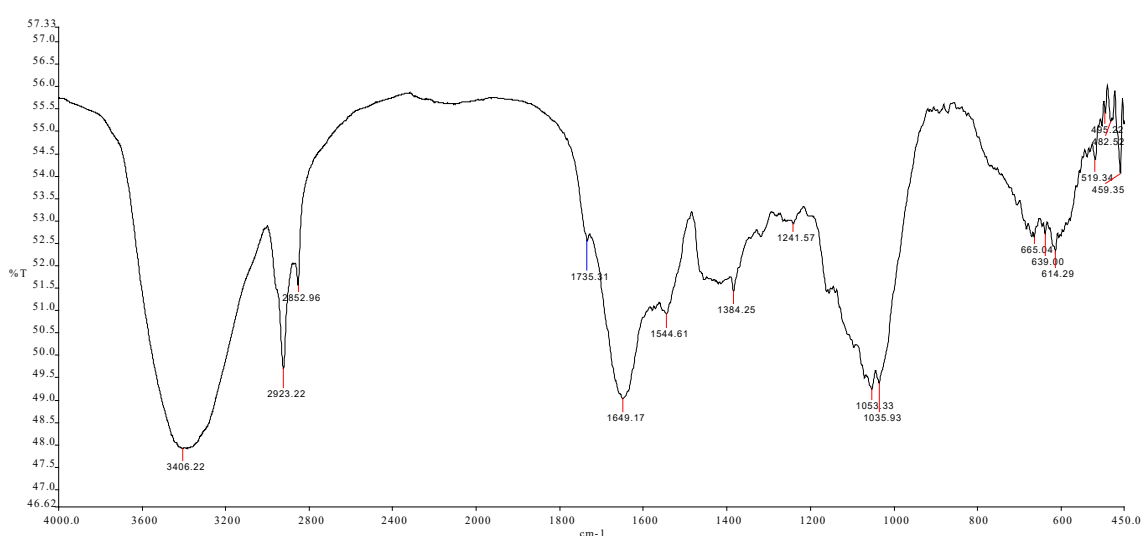
fibrous texture [17]. A noticeable change is observed on the surface morphology of CL-75 before and after dye adsorption possibly due to filled up of sites of CL-75 by BG dye and thereby the surface heterogeneity is diminished.

Table 1 Characteristics properties of CL-75.

Moisture	10.02 %
Bulk density	0.45 g/cm ³
Ash	1.2 %
Point of zero charge (pzc)	6.45
Particle charge	485 - 510 millivolt
Particle charge demand	(-)0.125 millivolt
Total pore volume	1.516×10 ⁻⁰² cc/g
Average pore radius	18.9479 Å
BET surface area	16.003 m ² /g



(a)



(b)

Figure 1 (a) FTIR spectra of CL-75 before adsorption and (b) FTIR spectra of CL-75 after adsorption.

Effect of parameters on adsorption

Initial concentration

The rate of adsorption depends on the initial concentration of the adsorbate, which is an important factor for effective adsorption [18]. The removal percentage and adsorption capacity of BG have been investigated in the range of 10.0 to 100.0 mg/L. The uptake capacity also increases steadily from 9.78 to 81.62 mg/g with an increase in the initial dye concentration due to the transfer of BG to the adsorbent surface by migration at low concentration of dye solution (**Figure 3**) [19]. As the concentration of BG increases the removal percentage decreases which may be due to the saturation of CL-75 sites or possible repulsive forces between adsorbed layers and remaining bulk molecules. The removal percentage of BG decreases from 97.0 to 83.0 % with the increase of the dye concentration (10.0 – 100.0 mg/L).

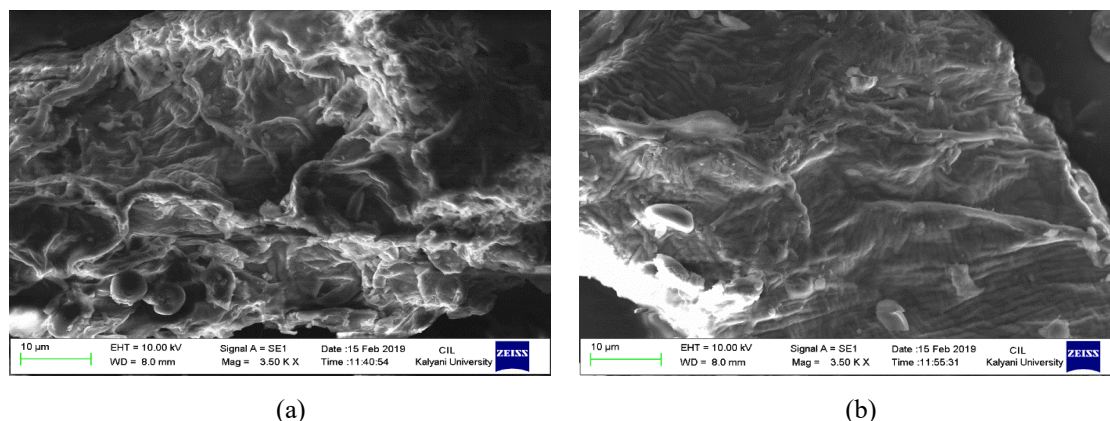


Figure 2 (a) SEM image of CL-75 before adsorption and (b) SEM image of CL-75 after adsorption.

Contact time

During adsorption of BG dye (20.0 mg/L) with CL-75 (50.0 mg), the contact time is varied (5, 10, 20, 30, 60, 90, 120, 150, 180, 210 and 240 min) at room temperature (**Figure 4**). The plot of %R vs. time indicates that the removal percentage of BG dye steadily increases (up to 90 min) and a smooth and continuous curve pattern is observed probably due to the mono-layer adsorption of BG dye on the surface of CL-75. The adsorption rate is rapid and reaches equilibrium within 120 min. It also signifies that a strong interaction exists between CL-75 and the BG dye in the aqueous environment.

pH

A series of experiments have been carried out with the BG dye (50.0 mg/L, pKa: 4.93 and 2.62) and CL-75 (50.0 mg). The pH of the medium is adjusted by adding 0.1 (N) HCl and 0.1 (N) NaOH solutions. The adsorption uptake capacity and the removal efficiency increase up to pH 7.0 (**Figure 5**). The values are found to be 18.38 mg/g & 91.88 % at pH 2.0 and 19.48 mg/g & 97.4 % at pH 7.0 respectively. The average values of uptake capacity and removal efficiency are 19.62 mg/g & 98.1 % respectively in the pH range 7.0 to 12.0. In the range of $\text{pH} < \text{pH}_{\text{PZC}}$, the electrostatic interactions are repulsive as positively charged BG molecules repel the positive charged CL-75 and removal percentages are comparatively low. The opposite trend is noticed in the range of $\text{pH} > \text{pH}_{\text{PZC}}$, where the electrostatic interactions are attractive which favours the adsorption between BG molecules and CL-75 [15]. The uptake capacity as well as the removal percentage reaches maximum (19.78 mg/g and 98.92 %) at pH 12.0 probably due to strong electrostatic interaction between positively charged BG dye and negatively charged CL-75 [20,21].

Dosages

In order to optimize the dose of the adsorbent, series of experiments have been performed with varied dosages of CL-75 (25, 50, 75, 100 mg; 50.0 mL) in aqueous solution of BG (50 mg/L) for 2.0 h. The removal percentages vary from 90 to 96 % (93.43, 96.65, 90.2 and 91.32 %) with the variation of dosages. The rate of dye adsorption increases with increasing concentrations of adsorbent and reached a constant value (50.0 mg) at equilibrium (**Figure 6**). At the dose greater than 50 mg of CL-75, the removal percentage of BG decreases possibly due to a dynamic equilibrium exists between dye molecules present at the surface and at the solution.

Adsorption kinetic study

The study of adsorption kinetics provides valuable information on reaction mechanisms. It also predicts the time at which the adsorbate is removed effectively from the solution phase [22]. In this study, three important kinetic models, viz. pseudo-first order, pseudo-second order, Weber and Morris intraparticle diffusion model [23] have been investigated with BG dye (20.0 mg/L), at different temperatures (30 and 40 °C) with constant stirring speed (120.0 rpm).

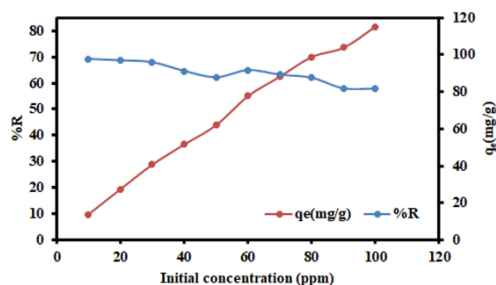


Figure 3 Effect of Initial concentration.

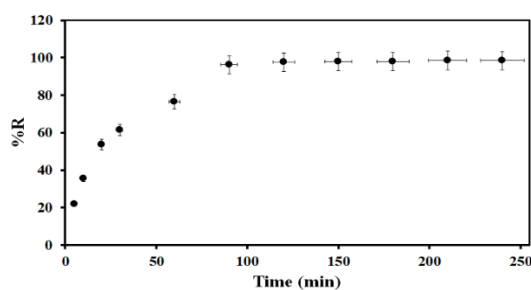


Figure 4 Effect of contact time.

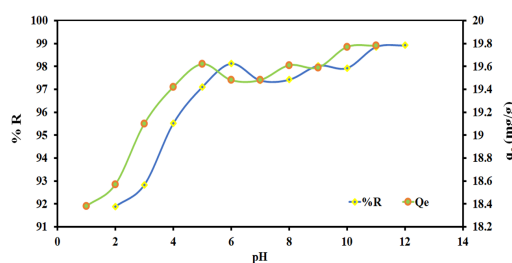


Figure 5 Effect of pH.

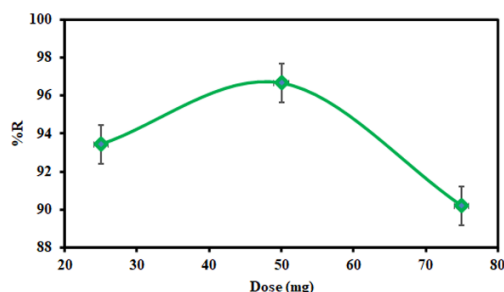


Figure 6 Effect of dosages.

The experiment shows an excellent linearity with high correlation co-efficient values ($R^2 = 0.99$, 0.98) in the case of pseudo-2nd order kinetic model in comparison to pseudo-1st order ($R^2 = 0.32$, 0.54) and intraparticle diffusion ($R^2 = 0.78$, 0.87) models. In the case of intraparticle diffusion model, the value

of diffusion coefficient, k_i , is also calculated (**Table 2**). It is clear from the plot of q_t vs $t^{0.5}$ that the intraparticle diffusion is involved in the adsorption process but it is not only the rate determining step.

Table 2 Parameters of kinetics studies for adsorption of BG on CL-75.

Temp (°C)	Pseudo-2 nd order kinetic				Pseudo-1 st -order kinetic model			Intra particle diffusion model	
	q_e	K_2	h	R^2	q_e	K_1	R^2	k_i	R^2
30	19.38	0.0417	15.67	0.99	1.47	0.0086	0.32	0.124	0.779
40	19.76	0.0311	12.151	0.98	2.2	0.0094	0.54	0.091	0.868

Note: The initial adsorption rate (h), the equilibrium adsorption capacity (q_e) and the 2nd order constants (K_2) can be determined experimentally (**Table 2**).

Adsorption isotherm study

The adsorption isotherm study provides relevant information regarding the interaction of sorbent and liquid phase under equilibrium condition at a certain temperature [16,24]. Several isotherm models are available based on some assumptions. Among various models, Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin are studied here (**Table 3**).

The Langmuir isotherm model is based on the assumption that the sorption method takes place on the homogeneous surface, i.e., sorption method is monolayer in nature. The dimensionless separation factor of Langmuir model, R_L , (Eq. (3)) offers important information about the shape of isotherm by interpreting as follows:

$$R_L = 1/(1 + bC_0) \quad (3)$$

$$R_L > 1.0 \quad \text{Unfavourable}$$

$$R_L = 1.0 \quad \text{Linear}$$

$$1 > R_L > 0 \quad \text{Favourable}$$

$$R_L = 0 \quad \text{Irreversible}$$

The Freundlich model provides information about the surface heterogeneity, distribution sites of CL-75 and energy of adsorption. The values of $1/n$ mostly predict the state of heterogeneity of surface by ranging between 0 and 1.0. The surface heterogeneity becomes predominating as it tends to 0 [16].

The interaction between the dye molecule and the bio-adsorbent may also be predicted by the Temkin model which is based totally on the assumption that the free energy of sorption is a characteristic feature of the surface coverage. The β value in D-R model provides [16] the mean free energy, E (kJ/mol) of adsorption per molecule of the adsorbate during the transformation from the solid surface from infinity in the bulk of the solution which is expressed as follows:

$$E = 1/\sqrt{2\beta} \quad (4)$$

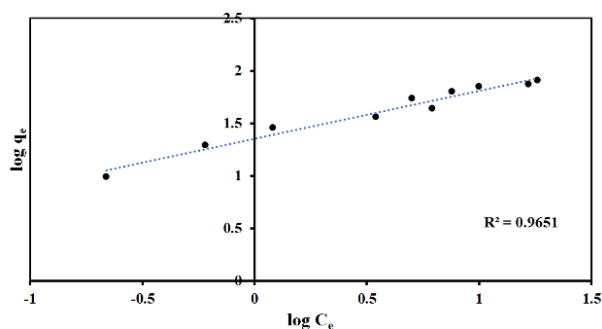
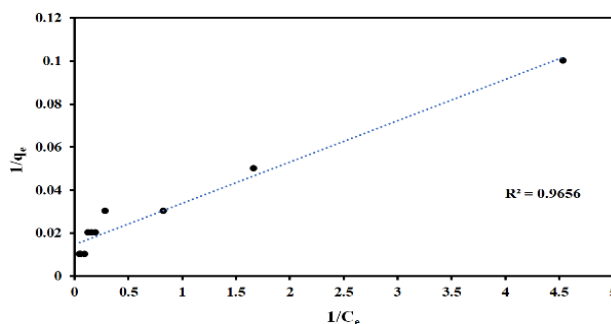
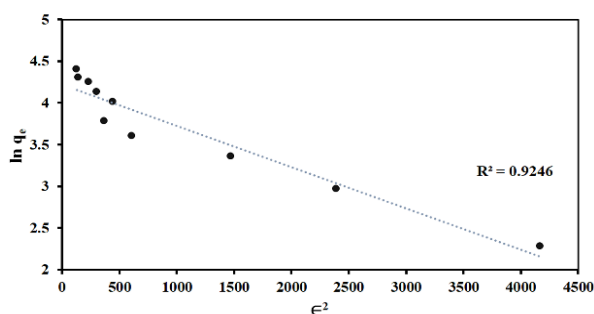
The parameters obtained from four isotherms models by linear fitting are given in the **Table 3**. Isotherm plots are given in **Figures 7 - 10**.

In case of Langmuir and Freundlich models, the relative high values of correlation coefficient satisfactorily explain the adsorption phenomena of BG dye onto CL-75. The maximum adsorption capacity (q_{\max}) of BG onto CL-75 is found to be 68.49 mg/g for the Langmuir model. The degree of adsorption affinity, b , indicates a stronger affinity between BG and CL-75. The R_L (0.02) value lies between 0 and 1.0 which indicates that the Langmuir isotherm is favourable. In the Freundlich model, the $1/n$ value is 0.4562, which also suggests a stronger adsorption between adsorbate and adsorbent molecules. The lesser the $1/n$ value the greater the expected heterogeneity. As the n value (2.192) is found to lie between 1.0 and 10.0, the sorption process is a favourable one [25].

Table 3 Different isotherm models and their parameters for adsorption of BG on CL-75.

Sl. No.	Isotherm Model	Linear form	R ²	Co-efficient
1.	Freundlich	$\log q_e = \log K_F + \frac{1}{n} \log C_e$	0.9651	$n = 2.192, K_F = 22.7$
2.	Langmuir	$\frac{1}{q_e} = \frac{1}{b q_0 C_e} + \frac{1}{q_0}$	0.9656	$b = 0.76, q_{\max} = 68.49$
3.	Dubinin-Radushkevich	$\ln q_e = \ln q_s - \beta \epsilon^2$	0.9246	$\beta = 0.0005, q_s = 67.67, E = 31.65$
4.	Temkin	$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e$	0.9202	$b_T = 154.83, A_T = 5.58, B = 16.002$

Where C_e is the equilibrium liquid-phase concentrations of the dye (mg/L), q_e and q_s are the equilibrium and maximum adsorbed amount of dye on CL-75 respectively (mg/g), b is the Langmuir equilibrium adsorption constant (L/mg) related to the free energy of adsorption, K_F is the Freundlich constant [(mg/g) (L/mg)^{1/n}] related to the strength of the adsorptive bond, and $1/n$ is the adsorption intensity factor or surface heterogeneity. In Temkin isotherm model RT/b_T corresponds to B which is related to the heat of adsorption and T is the temperature (K), R is the ideal gas constant (8.314 J/mol/K), and A is the equilibrium binding constant (L/min) and b_T is also a constant. In Dubinin-Radushkevich isotherm model q_s is named as theoretical saturation capacity (mol/g), β is a constant corresponds to the mean free energy of adsorption per mole of the adsorbate (mol²/J²), ϵ is the Polanyi potential [23].

**Figure 7** Freundlich isotherm for adsorption of BG dye on CL-75.**Figure 8** Langmuir isotherm for adsorption of BG dye on CL-75.**Figure 9** Dubinin-Radushkevich isotherm for adsorption of BG dye on CL-75.

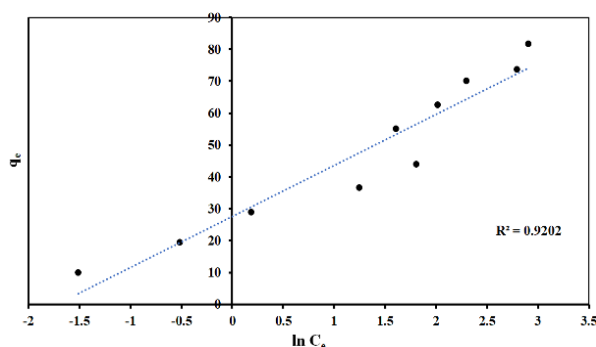


Figure 10 Temkin isotherm for adsorption of BG dye on CL-75.

The Gibb's free energy change is negative. The positive changes in enthalpy and entropy values further supported the spontaneity of the process (**Table 4**).

Table 4 Thermodynamic parameters for adsorption of BG on CL-75.

Temp (K)	ΔG (KJ/mol)	ΔH (KJ/mol)	ΔS (KJ/mol)
293	−82.44	20.01	0.011
303	−86.87		
313	−97.61		

Optimization study by Taguchi modelling

The Taguchi model, an effective tool of Design of Experiment (DOE), conducts series of experiments with minimum resources. In this study, interactions of three parameters (dose, concentration and time) with q_e are chosen. L9 orthogonal array (3×3) has been applied with nine experiments of three levels and the larger value is better in this experiment (**Table 5**) [25]. The S/N ratio is obtained from the following Eq. (5).

$$S/N = -10 \log \frac{\sum_{i=1}^n \frac{1}{y_i^2}}{n} \quad (5)$$

where, y_i is the estimated value of adsorption capacity and n is the number of replica of experiments under same conditions.

Table 5 Taguchi L9 orthogonal array design for removal of BG dye in the aqueous medium.

Sl. No.	Dose (gm)	Conc. (ppm)	Time (min)	Dose (gm)	Conc. (ppm)	Time (min)	q_e (mg/g)	S/N ratio
1	0.05	25.0	30.0	1	1	1	20.19	26.102
2	0.05	50.0	60.0	1	2	2	46.25	33.30
3	0.05	75.0	120.0	1	3	3	71.95	37.141
4	0.01	25.0	60.0	2	1	2	104.9	40.416
5	0.01	50.0	120.0	2	2	3	130.35	47.248
6	0.01	75.0	30.0	2	3	1	92.95	39.365
7	0.1	25.0	120.0	3	1	3	11.845	21.471
8	0.1	50.0	30.0	3	2	1	22.5	27.044
9	0.1	75.0	60.0	3	3	2	33.385	30.471

Where, dose of CL-75, conc. of BG dye and time of contact are considered.

It is revealed that adsorption capacities of BG dye vary from 11.845 to 130.35 mg/g obtained by combination of several factors.

Table 6 Response table for signal to noise ratio.

Level	Dose	Concentration	Time
1	42.34	29.33	30.84
2	32.18	35.86	34.76
3	26.33	35.66	35.29
Delta	16.01	6.53	4.45
Rank	1	2	3

Table 7 Response table for means.

Level	Dose	Concentration	Time
1	142.73	45.65	45.21
2	46.13	99.70	61.51
3	22.58	66.10	104.71
Delta	120.16	54.06	59.5
Rank	1	3	2

It is clear from the main effect plot (**Table 7**) that q_e value decreases with increasing dose and increases with increasing time. Initially it increases with concentration in the range 25.0 to 50.0 ppm and then decreases. The S/N ratio plot for each level of several factors indicate that the maximum variation takes place with the dose of CL-75 followed by concentration and time. In this perspective, dose has the most significant effect for highest adsorption capacity (**Table 6**).

Ranking of parameters based on S/N ratio is Dose > Concentration > Time means that dose affects q_e at highest level and time at lowest level (**Table 6**). The role of various parameters indicates that interactions between dose, concentration and time ranges from 0.05 - 0.1 g, 25.0 - 50.0 mg/L and 30.0 - 60.0 min are best possible ranges of parameters for experimental studies.

The ANOVA analysis has been performed to predict the statistical significance of variable parameters. During this analysis, the greater value of F ratio indicates major contribution of the variable or factor. ANOVA results (**Table 8**) reveal that dose has the higher influence on the uptake of dye followed by time and concentration. Hence, the order of several factors is Dose > Time > Concentration.

Table 8 Analysis of Variance for q_e on BG dye.

Source	DF	MS	SS	F	p
Concentration	2	4469	2235	0.41	0.683
Time	2	5673	2836	0.53	0.611
Dose	2	24325	12162	5.55	0.043

Note: DF is degree of freedom, MS is mean square, SS is sum of square and F is F-ratio.

Conclusions

In the present work, the biomass derived from coriander leaf has been investigated as a potential adsorbent for the removal of brilliant green dye in the aqueous environment. The effects of pH, dose, concentration of dye and time of contact have been investigated. The uptake capacities and the removal percentages are found to be maximum at $\text{pH} > \text{pH}_{\text{PZC}}$ due to strong electrostatic interaction. The adsorption process follows Freundlich and Langmuir isotherms (q_{max} - 68.49 mg/g) and fit with pseudo-2nd order kinetic model based on higher regression value. The Gibb's free energy change is negative which also favours the spontaneity of the process. The Freundlich constant, n , suggests a strong interaction between the dye and the adsorbent. Taguchi experimental design with L9 orthogonal array has been studied for the interaction of parameters (dose, concentration and time) during the removal of dye in

the batch mode considering the highest adsorption uptake capacity. Among three factors, dose has the highest influence on the uptake of dye followed by time and concentration, and further supported by the ANOVA analysis (Dose > Time > Concentration). Thus, the adsorbent derived from the coriander leaf might be helpful for the sequestration of Brilliant green dye from real samples as well as industrial effluents.

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