Removal of brilliant green dye from waste water using zinc peroxide-charcoal composite

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Abstract

The present work has been envisaged to develop an efficient process for adsorptive decontamination of Brilliant green (BG) dye from waste water at room temperature. In recent past, various natural as well as advanced materials and composites have been reported to remove dyes from water. But these materials have certain limitations like time consumption, lower adsorption capacity and residual toxicity etc. To overcome these limitations, zinc peroxide-charcoal composite (ZnO₂-C) has been synthesized by wet chemical route. The proposed composite efficiently decontaminates BG from waste water within 25 minute in wide pH range (2-9). The spectral and microscopic studies have been done for ZnO₂-C composite before and after adsorption of BG to know the adsorption behavior of proposed material. Various parameters like pH, adsorbent dose, contact time and dye concentration were optimized by the batch sorption experiment to determine maximum adsorption capacity of ZnO₂-C. The adsorption capacity of the composite was found to be 156.1 mg g⁻¹ which is better than several other adsorbents cited in literature in recent past. The experimental adsorption data follows second order kinetics and Freundlich adsorption isotherm. Copyright © 2017 VBRI Press.

Keywords: Adsorption; brilliant green; zinc peroxide-charcoal composite.

Introduction

Dyes are commonly utilized in various industries like textile, leather, cosmetics, paper, pulp, tannery etc. for coloring of various materials. These industries generate huge amount of effluent water which gets discharged into surface or river water without treatment. Worldwide textile industries require large amount of dyes in comparison to other industries [1]. It has been estimated that more than 1000 tones/ year of dyes are discharged into surface water which causes severe health hazard to human and aquatic lives [2]. Depending on the exposure time and dye concentration, it can cause cancer, mutation, skin irritation and also affects cardiovascular system, gastrointestinal system and central nervous system etc [3]. There are various dyes available in the market some are Malachite green, Congo red, Methylene blue, Methyl orange, Crystal violet and Bromophenol blue. Out of these dyes Brilliant green most commonly used in industries on a large scale.

BG is a cationic dye containing triarylmethane group. It is most commonly utilized in many textile and paper industries for the manufacturing of cover paper, dyeing



Fig. 1. Structure of Brilliant green dye.

clothes etc. **[4]**. As per literature around 1 kg of BG dye is utilized for the production of 1 ton of paper. Apart from its industrial usage it is also utilized in large number of biological applications such as in biological staining,

dermatology, medicines and poultry farms as food additive to prevent spread of mold, intestinal fungus and parasites[5]. BG causes irritation to eyes and may even lead to eyes damage. If inhaled even in low quantity, it can cause serious problems like coughing, shortness of breath etc. in the respiratory tract. It can also cause problems like nausea, diarrhea and vomiting if swallowed in gastrointestinal tract [6]. Upon disintegration, it leads to the formation of harmful compounds such as oxides of nitrogen, carbon and sulfur [7]. Thus it becomes very important to remove this hazardous dye from water.

Keeping the toxicological effects of dye bearing effluents, there is an urgent need to develop a robust technique for their removal up to desired concentration levels. In this regard, various processes have been applied for their removal which includes chemical oxidation[8], coagulation[9], adsorption[10], membrane separation[11], reverse osmosis[12], electrochemical treatments[13], photo degradation[14], and catalytic microbial degradation[15]. However these processes have their own limitations like chemical oxidation requires oxidants, coagulation is not effective for soluble dyes; membrane separation, reverse osmosis, electrochemical treatments are expensive; microbial degradation removes dye upto some extent and time consuming; photo catalytic degradation requires UV light with photo catalysts. In recent years, various adsorbents like chitosan[16], fly ash[17], clay[18], carbon nanotubes[19], maghemite[20], carbon/iron oxide nano-composite [21], activated carbon[22] etc. have been reported for removal of dyes from waste water. However these adsorbents have certain drawbacks regarding removal capacity, cost, time, regeneration etc.

The precedent work highlights the efficiency of zinc peroxide-charcoal composite (ZnO_2-C) synthesized by wet chemical route for the efficient removal of BG from waste water at room temperature. The removal of BG has been studied under optimized condition of pH, adsorbent dose, adsorbate concentration and time to obtain maximum removal efficiency.

Experimental

Materials and methods

All chemicals like zinc acetate dihydrate, ammonium hydroxide, polyvinylpyrrolidone (PVP), hvdrogen peroxide were of analytical grade and procured from E. Merck India. Charcoal powder and Brilliant green dye was procured from BDH chemicals Ltd and Loba Chemie Pvt. Ltd respectively. A 1000 mg L⁻¹ stock solution of BG was prepared by dissolving 1g of dye powder in 1000 ml of de-ionized water. This solution was further diluted with de-ionized water to the desired concentrations for the experimental work. The pH of the solution was adjusted with 0.1M NaOH or HCl solution using Orion make pH meter. The concentration of BG in the solutions was determined by measuring the absorbance at 620 nm using Hitachi make UV-Vis. Spectrophotometer.

Preparation of ZnO₂-C

For the synthesis of ZnO₂-C composite, 20g of zinc acetate was dissolved in minimum amount of water. Then ammonium hydroxide solution was added until a clear solution was obtained upon constant stirring (500 rpm) at 60-70 °C. Then 2g of PVP was added to control the particle size and mixed thoroughly with constant stirring. After 1h, 30g of charcoal powder was added to the solution. Further 50 mL of 30% hydrogen peroxide was added to the solution drop by drop. Black colored precipitate was formed which was centrifuged at 10,000 rpm and washed several times with de-ionized water and finally dried in an oven at 105 °C.

Characterization

The synthesized ZnO₂-C before and after adsorption of BG dye was characterized for its phases by Bruker make AXS D8 X-ray Diffractometer (XRD) using CuK α radiation with a graphite monochromator in the diffracted beam. The shape and size of ZnO₂-C before and after adsorption of dye was characterized using FEI, Netherland make high resolution Transmission Electron Microscope (HR-TEM); model F-30 G2 STWIN. The functional groups of ZnO₂-C before and after adsorption of dye was characterized using Agilent make Cary 630 Fourier transform infrared (FTIR) spectrophotometer.

Batch adsorption studies

Batch adsorption experiments were carried out to determine the maximum uptake of adsorbate (BG dye) by adsorbent (ZnO₂-C). For this, 0.05 g of ZnO₂-C was added to a 25 mL dye solution (10 mg L^{-1}) and the solution was ultrasonicated stirred for 30 minutes. Further the solution was centrifuged (8000 rpm) to separate ZnO₂-C from the solution. The concentration of the residual dye in solution was analyzed by UV-Vis spectrophotometer. The same procedure was repeated at varying adsorption parameters (pH, adsorbent dose, contact time and dye concentration) to optimize maximum uptake of dye by ZnO₂-C. Equation (1) and (2) were used to determine % removal and adsorption capacity.

% removal =
$$\frac{\left(C_{i} - C_{f}\right) \times 100}{C_{i}}$$
 (1)
Adsorption capacity $\left(q_{e}\right) = \frac{\left(C_{i} - C_{f}\right) \times V}{m}$ (2)

Where, C_i and C_f (mg L⁻¹) are the initial and final concentration of BG dye solution, *V* is volume (mL) of solution used and *m* is the mass (g) of adsorbent. The schematic describing adsorption of BG by ZnO₂-C has been presented in **Fig. 2.**



Fig. 2. Schematic diagram for adsorption of BG by ZnO₂-C.

Results and discussion

Characterization

XRD analysis

The phase analysis of pure charcoal, ZnO_2 -C and BG adsorbed ZnO_2 -C (ZnO_2 -C-BG) were carried out by XRD in 2 θ range from 20° to 80°. The XRD pattern of the three materials has been presented in **Fig. 3** (a). Pure charcoal shows a sharp diffraction peak at 26.1° corresponding to (002) plane, ZnO_2 -C shows other characteristic peaks at 31.6° (111), 36.6° (200) of ZnO_2 in addition to 26.1° corresponding to (002) plane of charcoal. No additional peak was observed in XRD pattern of ZnO_2 -C-BG which indicates the adsorption of BG did not alter the crystal structure of ZnO_2 -C.



Fig. 3. (a) XRD and (b) FTIR pattern of (i) Pure Charcoal (ii) ZnO₂-C and (iii) ZnO₂-C-BG.

FTIR analysis

FTIR analysis is a useful tool in identifying the functional groups present over the adsorbent. **Fig. 3** (b) shows the FTIR spectra of (i) pure charcoal, (ii) ZnO_2 -C and (iii) ZnO_2 -C-BG in the wavelength range of 4000-400 cm⁻¹. The exhibition of peak at 1396cm⁻¹ in pure charcoal is assigned to C=C stretching. In FTIR spectrum of ZnO_2 -C, another peak originated at 430 cm⁻¹ is due to Zn-O stretching. The spectra of ZnO_2 -C-BG show several characteristic peaks in addition to those of C-C and Zn-O stretching which indicates the adsorption of BG. The peaks at 1493 and 1093 cm⁻¹ are assigned to C=C and C-N (tertiary amine) stretching respectively.

TEM analysis

For detail analysis of adsorption process of BG over ZnO₂-C, TEM study of pure charcoal, ZnO₂-C and ZnO₂-C-BG were carried out. Fig.4 (a-c) shows the TEM micrograph of pure charcoal, ZnO2-C and ZnO2-C-BG respectively. The micrograph shows the remarkable changes in pure charcoal after forming composite with ZnO₂ (FIG.4(b)) and further adsorption of BG over ZnO₂-C as shown in **Flig.4(c)**. In Fig. 4(b), ZnO₂ particles seem to be uniformly distributed throughout the charcoal matrix whereas Fig.4(c) clearly shows the adsorption of dye molecules over ZnO₂-C composite surface. Left and right insets in Fig.4 (a-c) are the diffraction pattern and high resolution images of pure charcoal, ZnO₂-C and ZnO₂-C-BG respectively. Diffraction pattern clear reveal that ZnO₂-C and ZnO₂-C-BG have lattice spacing d=2.81 Å and d=2.44 Å correspond to 111 and 200 plane of ZnO_2 whereas diffraction of pure charcoal shows the amorphous nature. High resolution images also confirm the d=2.44 Å correspond to 200 plane of ZnO₂. These results are in good agreement with XRD result.



Fig. 4. TEM micrograph of (a) Pure Charcoal (b) ZnO_2 -C and (c) ZnO_2 -C-BG*pH variation*.

In order to examine the effect of pH on the removal of BG dye by ZnO_2 -C, experiments were performed within a pH range of 2-9 as shown in **Fig. 5** (**a**). For this, ZnO_2 -C (0.05 g) was added to 25 ml dye solution with an initial concentration of 10 mg L⁻¹. The figure reveals that proposed composite is highly efficient in wide pH range (2-9) and there is no need of any acidic or basic medium for functioning of the composite.

Effect of adsorbent dose

The effect of adsorbent dose on % removal of BG dye was studied by varying the amount of ZnO_2 -C from 0.005 to 0.05g keeping pH, volume and dye concentration same as in pH variation study. It is clear from **Fig. 5 (b)** that the % removal of the dye increased with an increase in the adsorbent dose. It is because of the fact that increase in adsorbent dose eventually increased the number of sites available over adsorbent for adsorption of dye which resulted into enhancement of removal efficiency. The maximum adsorption was observed at 0.04g of ZnO_2 -C.

Effect of contact time

The contact time between the BG dye and the ZnO_2 -C was varied from 5 to 40 min. The percent removal of the dye was calculated for each time interval. The results for % removal of BG vs. contact time are shown in the **Fig. 5 (c).** It can be concluded that with increase in time, there is an increase in removal efficiency; it is because of the fact that desired time is required for maximum interaction between the adsorbent and dye. Thus ZnO_2 -C should be in contact with BG for 25 min in order to obtain maximum adsorption.

Effect of dye concentration

The effect of dye concentration was studied by varying the concentration of the dye solution from $5-250 \text{ mg L}^{-1}$. For this 0.04 g of adsorbent was taken in 25 ml of dye solution and percent removal was calculated after 25 min of investigation. From **Fig. 5** (d) it can be concluded that the % removal of dye decreases with increase in dye concentration because of the saturation of the active sites of ZnO₂-C after adsorption of certain concentration of BG.



Fig. 5. Effect of (a) pH (b) Adsorbent dose (c) Time (d) Dye concentration on % removal of BG.

Kinetics study

Adsorption kinetics study provides useful information about adsorption mechanism. For this, the adsorption experiments were carried out at different time interval for 10 mg L⁻¹ concentration of BG dye solution and the data obtained were fitted two kinetic models, Pseudo first order [23] and pseudo second order [24] kinetic models. The linear form of pseudo first order model has been presented by eqn. (3).

$$\ln\left(q_{e} - q_{t}\right) = \ln q_{e} - K_{1}t \tag{3}$$

Where, q_e and q_t is the amount of BG adsorbed (mg g⁻¹) at equilibrium and at time't' respectively, K_1 (min⁻¹) is the pseudo first order rate constant. The plot of ln (q_e - q_t) against't' gives a linear curve as presented in **Fig. 6 (a)**. The value of K_1 and q_e were obtained from slope and intercept of same curve.

The linear form of pseudo second order model is represented by eqn. (4)

$$t/q_{t} = 1/K_{2}q_{e}^{2} + t/q_{e}$$
(4)

Where, K_2 (g mg⁻¹ min⁻¹) is the pseudo second order rate constant. The plot of t/q_t against t gives a linear curve as presented in **Fig. 6(b)**. The value of q_e and K_2 were obtained from slope and intercept of same curve.

From **Fig. 6(a)**, the value of K_1 and q_e were calculated to be 0.392 min⁻¹ and 1.657 mg g⁻¹. Similarly for second order kinetics as presented in **Fig. 6(b)**, the values of q_e and K_2 calculated from slope and intercept were found to be 6.289 mg g⁻¹ and 1.404 g mg⁻¹ min⁻¹. The value of correlation coefficient (R²) for pseudo second order kinetic is found to be higher in comparison to pseudo first order kinetics which confirms that experimental adsorption data was best fitted to second order kinetic model and suggests that chemisorption is the rate limiting step during the adsorption process.

Isotherms study

Adsorption Isotherm studies describe how the adsorbate interacts with adsorbent. It also gives the maximum adsorption capacity of adsorbent. For this, the adsorption experiments were carried out at different concentration of BG from 5-250 mg L⁻¹ and the data obtained were fitted to Isotherm models. Langmuir[**25**] and Freundlich[**26**] are the two most commonly used isotherms models to describe the interaction between adsorbent and adsorbate. Langmuir isotherm indicates monolayer adsorption of adsorbate over homogenous surface of adsorbent. It assumes that surface of adsorbent carries fixed number of sites having same energy. Each site can hold only one adsorbate without any interaction between adsorbed molecules. The linear form of Langmuir isotherm is given by following equation.

$$C_f/q_e = 1/q_m K_L + C_f/q_m \tag{5}$$

Where, $q_e (mg g^{-1})$ is the amount of adsorbate adsorbed at equilibrium, $C_f (mg L^{-1})$ is the equilibrium concentration of adsorbate, $q_m (mg g^{-1})$ is the maximum adsorption capacity of adsorbent and K_L (L mg⁻¹) is Langmuir constant. The plot of C_f/q_e against C_f gives a linear curve as presented in **Fig. 6** (c). The value of q_m and K_L were obtained from slope and intercept of same curve. The separation factor 'r' [27], which reveals feasibility of Langmuir isotherm, has been calculated using following equation.

$$r = 1/(1 + K_L C_o)$$
(6)

Where $C_o(mg L^{-1})$ is the initial concentrations of adsorbate, r < 1 indicates favorable adsorption and r > 1 as unfavorable process.

Freundlich isotherm suggests multilayer adsorption of adsorbate over heterogeneous surface of adsorbent. It assumes infinite number of vacant adsorbent sites having different energy of adsorption. The linear form of Freundlich isotherm is given by eqn. (7)

 $lnq_e = ln K_F + 1/n lnC_f$

Where $K_F [mg g^{-1} (L mg^{-1})^{1/n}]$ and 1/n are Freundlich constants indicating adsorption capacity and adsorption intensity respectively[**28**]. The plot of lnq_e against lnC_f gives a linear curve as presented in **Fig. 6(d)**. The values of K_F and 1/n were calculated from the intercept and slope of same curve.



Fig. 6 (a) Pseudo first order (b) Pseudo second order (c) Langmuir isotherm and (d) Freundlich isotherm plot for BG adsorption over ZnO_2 -C.

The value of Langmuir and Freundlich adsorption isotherm parameters calculated from slope and intercept of both linear curves are summarized in **Table 1**.

Table 1. Langmuir and Freundlich isotherm parameters for adsorption of BG by ZnO_2 -C.

Dye	Langmuir Isotherm Parameter			Freundlich Isotherm Parameter		
	$q_m(mg g^{-1})$	K _L (L mg ⁻¹)	\mathbb{R}^2	$\frac{K_{F} (mg g^{-1}}{(L mg^{-1})^{1/n}})$	N	\mathbb{R}^2
BG	142.85	6.99	0.88	356.74	1.66	0.99

From the results of both isotherms, it has been observed that Freundlich adsorption isotherm model was best fitted to adsorption data in terms of higher R^2 than Langmuir model. The q_m calculated from Langmuir isotherm is found to be 142.85 mg g⁻¹ whereas the experimental adsorption capacity was found to be 156.1 mg g⁻¹ for 250 mg L⁻¹ concentration of BG. The applicability of Freundlich isotherm indicates the multilayer adsorption of BG over heterogeneous surface of ZnO₂-C. The adsorption capacity of several adsorbent which has been utilized for BG decontamination are given in **Table-2**.

S. N.	Adsorbent	pН	Time	Adsorption capacity (mg g ⁻¹)	References
1.	Peanut shell	9	60 min	19.92	[29]
2.	Rambutan peels	2	24 h	9.64	[30]
3.	Sawdust	2.9	3h	58.47	[31]
4.	Acorn based adsorbent	6- 10	30 min	2.1	[32]
5.	Saklikent/) mud	1-2	1h	9.2	[33]
6.	PET bottles	6	48h	33	[34]
7.	ZnO ₂ -C	2-9	25 min	156.1	Present work

Table 2. Adsorption capacity of different adsorbents for BG dye.

Reusability study

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Reusability study reveals the economical utility of an adsorbent. It helps in reducing resources, energy, synthesis cost and time. The adsorption reusability of proposed composite was tested for 10 mg L⁻¹ solution of BG. For this, the BG adsorbed composite was dried and used for further adsorption of same concentration of BG. The percent removal of BG was calculated with same time and adsorbent dose and found to be 97.4 %. The same composite was used again for second adsorption cycle with same volume and concentration of BG. The percent removal decreases to 92.2%. The removal efficiency of composite for BG in third and fourth cycle decreases to 77.3 and 50.5% respectively. Thus it can be concluded that the reusability of ZnO₂-C for BG makes it economical and efficient adsorbent.

Conclusion

An efficient ZnO₂-C composite adsorbent has been synthesized by wet chemical route for the removal of BG from waste water. The extent of adsorption has been investigated for various operating parameters like; adsorbent dose, agitation time, pH and dye concentration. The adsorption of BG was also verified by XRD, FT-IR and TEM techniques. The proposed material works in wide range of pH (2-9), so there is no need to adjust the desired pH of the test water for functioning of adsorbent. The adsorption capacity of the proposed material found to be 156.1 mg g⁻¹ which is much higher than several other adsorbent reported in the literature. The adsorption capacity of composite material in first run was found to be excellent, where as in second, third and fourth run and adsorption capacity found to be better than several adsorbent given in table-2. The experimental adsorption data was fitted to kinetics and isotherm models to design the adsorption process. Adsorption kinetics follows pseudo second order kinetics. Isothermal modeling were studied which shows that this data fits best to Freundlich isotherm. The proposed material is cost effective and economical viable. Beside these for the synthesis of composite material there is no need of any sophisticated or costly instruments. So the purified water can be reused in the same industry.

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Author's contributions

Conceived the idea and plan: NS; Performed the experiments: SC, HU, MY; Data analysis: SC, HU, MY, DS, NS; Wrote the paper: SC. Authors have no competing financial interests.

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