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Studies on the removal of Brilliant Green dye using low cost agricultural waste

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The potential use of Sugarcane Bagasse (SB) as environment friendly and low-cost industrial waste for the exclusion of brilliant green (BG) dye from wastewater has been studied. For this purpose, the batch adsorption process is applied to find adsorption kinetic and thermodynamic data. The second-order pseudo-kinetic model and Langmuir isotherm are found well suitable to explain the adsorption process of BG onto SB. The highest adsorption capacity analyzed using the Langmuir isotherm is 24.32 mg/g at 50°C. The Gibbs free energy (ΔG) values were observed to be negative at all functioning temperatures, validating that the BG is positive, shows the higher randomness at the adsorbent-adsorbate interface. In general, the study results propose that SB can be used as a low-cost and environment friendly agro-industrial waste material for effective removal of BG dye.

Keywords: Sugarcane Bagasse, Brilliant Green, Adsorption, Langmuir, Kinetics

Industries such as carpet, paints, textile, pulp,and paper consume a tremendous quantity of water and therefore categories as water intensive industries. During their operation, these industries generate a substantial quantity of waste water, specifically wastewater containing synthetic dyes which are of great concern. The waste water containing dye is aesthetically and environmentally not acceptable due to high odour, colour, obstruction to penetration of light and high chemical oxygen demand (COD). Furthermore, due to the aromatic structure; thesedyes are very stable in nature, which leads to their slow degrade[1,2]. Synthetic dyes are also very toxic in nature and may have carcinogenic and mutagenic effects, which has a diverse effect on ecosystem health.

Therefore, there is a need of an urgent action to explore efficient, environmentally friendly and low cost methods for the removal of dyes form waste waters[2,3]. One of such dye which is mostly generated during the process of textile dyeing, paper printing, preparation of veterinary medicine, etc. is Brilliant Green (BG) dye which has adverse effects on the surrounding ecosystem[4,5]. It was observed that nearly 50% of used dyes were discharged directly in the receiving water streams without any treatment[6]. Therefore; there is a need to explore methods and techniques for efficient removal of BG from waste water containing a dye so as to protect the surrounding ecosystems.

Various physico-chemical methods of treatment such as oxidation, photocatalysis, electrochemical, coagulation and flocculation, microbial degradation, membrane separation, ion exchange,etc. are now being used to treat dyes waste water[1,7]. Amongst the aforesaid methods, adsorption has gained more attention because of its features such as efficiency effectiveness, ease of operation, minimum energy requirement, reuse capability, low cost, and simple design. Various studies havebeen done on use of natural and locally available adsorbents such as *Solanumtuberosum* peels, oxidized cactus fruit peel, neem leaves, acorn activated carbon, acid-activated watermelon rind, jackfruit peels, banana peel, tea leaves, mangosteen peels, etc. for the effective removal of BG from dye wastewater via adsorption[7]. However, scanty research has been done to explore the potential waste from plant residue specifically Sugarcane Bagasse (SB) for the effective removal of dye waste water.

The focus ofthe present study is to explore the use of SB a solid fibrous residue of sugarcane after juice extract for the effective removal of BG from aqueous solutions. The SB is available in a huge amount from the sugarcane industry, and mostly considered as waste and disposed of. The utilization of SB as an adsorbent in wastewater treatment serves duel purposes; firstly it will treat the dye containing wastewater and secondly it will help in reducing the waste burden in the industry and surrounding environment. In this regard; to use the SB as an adsorbent; the detail physicochemical characterization viz. proximate analysis, BET surface area, and FTIR were carried out before and after the adsorption. Furthermore; the effect of operating parameters namely adsorbent dose, initial concentration, contact time and the temperature were exploredin detail to optimize the adsorption process. Additionally, kinetic and thermodynamic parameters are determined by applying different models forthe batch adsorption process.

Experimental Section

Adsorbent preparation

SBwas collected from a local industry situated in Kadodara, Surat (Gujarat, India). The physical impurities of adsorbents were removed by washing several times with tap water followed by double-distilled water. The collected samples were thencut into small parts and were dried in a hot air oven at 70°C til achieving uniform weight. The samples were then crushed, andt he fine fraction was separated. The fine fraction of SB sample was stored in sealed bags and kept inside the desiccators for further use.

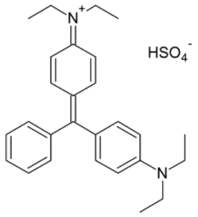
Chemicals

Exactly 1 g of BG (Merck, India) in 1 L of deionized water was dissolved, and a 1000 mg/L of the stock solution was prepared.The different concentrations for experiments were then prepared from the stock solution. The 0.1N NaOH/HCl (Fisher Scientific, USA) chemical was used to maintain the *p*H of the solution as and when required.During each experiment, fresh dilutions were made using double distilled water. The physical properties and chemical structure of BG are shownin Table 1.

Batch adsorption studies

During batch adsorption, exactly 1 g of dye in double distilled water was dissolved, and 1000 mg/L stock solution of BG was prepared. During experimentations, the other required concentrations from stock solution were prepared by subsequent dilutions. In each flask, 50 mL dye solution of specific concentration (15, 20, 50, 70, 100 mg/L)and adsorbent was mixed and kept in water bath shaker (Remi, RSB-12) at150 rpm at defined temperatures (30, 40, 50°C) and time. After every experimental study, the solutions were filtered using a What manfilter, andthe filtrate dye concentration was measured spectrophotometrically. The performed experiments wereanalyzed thrice, and the average values of the experiment were reported. The calculated standard deviation (%) was minimum and therefore not reported. The used adsorbent was collected, dried and stored in sealed bags for reuse in other applications.

Table 1 — Properties and characteristics of BG.

Common name	Brilliant green
Chemical structure	
Chemical formula	$C_{27}H_{33}N_2.HO_4S$
Molecular weight (g/mol)	482.64
Solubility (water g/100 mL)	100 g/L at 20 °C

The percentage adsorption and capacity of adsorption, i.e., q_e (mg/g) of the SB at equilibrium were considered using equations 1 and 2.

$$\text{Adsorption (\%)} = \frac{(C_0 - C_e)}{C_0} \times 100 \qquad \text{... (1)}$$

$$q_e = \frac{(C_0 - C_e)V}{w} \qquad \text{... (2)}$$

The experiments were performed for variable doses, concentrations, times, *p*H and temperatures. The equilibrium information obtained were applied to various models (i.e., kinetic, thermodynamic, and isothermal) to specify the best fit suitable model for the system.

Instrumentation and characterization

The standard procedurewas used for proximate analysis (IS 1350)[8]. The Brunauer–Emmett–Teller (BET) surface area of adsorbents was calculated using surface area analyzer (Smart Sorb 92/93). Fourier-transform infrared spectroscopy (FTIR) spectra of raw adsorbent and dye-loaded adsorbent were obtained using the KBr disk method (Thermo Scientific, Nicolet iS5). The BG (λ_{max}=624 nm) analysis was performed using UV–vis spectrophotometer (Shimadzu UV-1800). The *p*H of solutions was calculated using *p*H meter (Eutech Instrument model *p*H 2700).

Results and Discussion

Characterization of SB

Proximate analysis of SB

In the proximate analysis, the percentage of ash, moisture, dry matter and volatile matter of dried SB were determined. The results shown in Table 2 indicate that SB has high carbon and volatile matter and low ash content which confirm the organic nature of adsorbents.

BET surface area

During experiment 2.44 m^{2} g^{-1} was the observed value of the BET surface area of SB. The value of surface area shows the complexity during the operation related to the degassing of SB samples. Furthermore, the minimum surface area is a typical property of materials which is carbonaceous in nature[9,10].

FTIR analysis

The FTIR analysis was performed to explore the interactions of adsorbent and adsorbate. It is one of the important parameters to explore the functional group's presence on the adsorbent surface. Figure 1 shows the comparative FTIR analysis ofraw adsorbents and dye-loaded SB. FTIR spectrumrevelsthe availability of amino acids, amines, carboxylic acid, alkanes, alkyl halides, alcohol, and phenol in SB.

Table 2 — Proximate analysis of BP

Parameter	Value
Dry matter (%)	91.65
Moisture (%)	8.35
Ash (%)	3.26
Volatile matter (%)	86.37
Fixed Carbon (%)	1.17

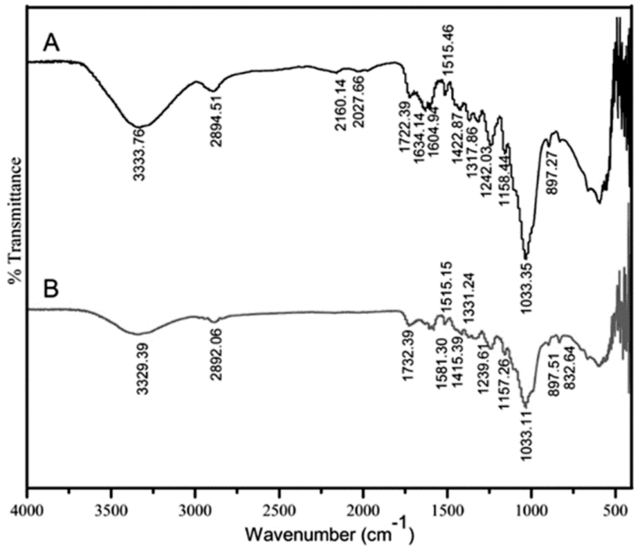


Fig. 1 — FITR Spectra of SB (A. Before adsorption; B. After adsorption)

The stretching absorption band obtained at 3333.76 cm^{-1} is assigned for NH or OH group in SB, after adsorption, it is shifted to 3329.39 cm^{-1}. The band obtained at 2894.51 cm^{-1} highlights vibrations of stretching of $-CH_3$ and or $-CH_2$ groups of carboxylic acid and its vibration of bending is at about 1317.86 cm^{-1}, after adsorption, it is shifted to 2892.06 cm^{-1} and 1331.24 cm^{-1}, respectively. The 1634.14 cm^{-1} peak shows the C=C stretch of an aromatic, alkene, or amino acids in SB, which is absent on the surface of SB after adsorption. The aldehyde carbonyl stretching bandis obtained at 1722.39 cm^{-1}, and it is shifted to 1732.39 cm^{-1} after adsorption. The presence of tertiary alcohol and or phenol was confirmed by peaks at 1242.03 cm^{-1} and 1033.35 cm^{-1}; Furthermore; primary amine and C–O stretch also displays minor shifts at 1033.11 cm^{-1} and 1239.61 cm^{-1} respectively.

Effect of operating parameters

Effect of adsorbent dosage

The effect of adsorbent dosage on BG removal was analysed by varying the amount of SB (0.1–1.2 mg) at a fixed initial concentration (50 mg/L). This solution mixture was kept for 180 min in water-bath shaker at 30°C. Results obtained are highlighted in Fig. 2. From the figure, it was observed that, with the increase in adsorbent dosage, the percentage ofadsorption increases because of the availability of more active sites during the initial stage of adsorption[11]. The adsorbent dosage further increase does not have any substantial change in the BG removal. Due to this reason, the adsorbent dosage of 0.9 g/50 mL was optimized for further studies.

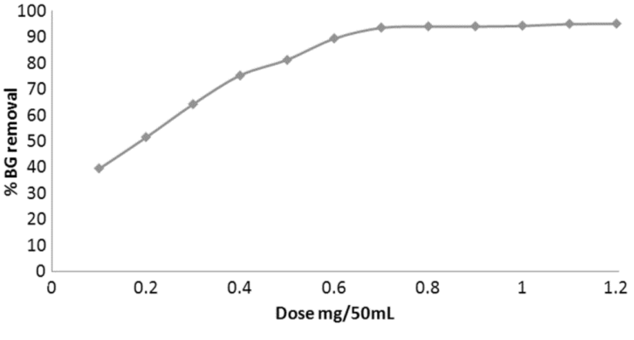


Fig. 2 — Effect of adsorbent dose on BG dye removal (C_0: 50 mg/L, temperature: 30°C; rpm: 150)

Effect of contact time

The contact time effect is an important factor for the wastewater treatment system design since it determined the equilibrium time. To calculate equilibrium time for BG adsorption process, the experiments were performed at different time intervals, for fixed concentration, temperature, and *p*H. The variation for removal of BG at various times duration for 50 mL of BG solution with adsorbent dosages of 0.9 g/50 mL at different initial concentrations (20-150 mg/L) and 30°C is highlighted in Fig. 3. From Figure 3; it was observed from that the maximum (≈90%) adsorption occurred during the first 60 min. After 60 min, the adsorption is constant; hence the time 70 min is considered as equilibrium time.

The adsorption capacity of the adsorbents increases as time increase.At the initial stages, the adsorbent stages are vacant do the adsorption rate was highest during the initial period[11].

*Effect of solution p*H

The structure and stability of dye molecules along with the functional group dissociation on the adsorbent are mostly solution *p*H driven. The *p*H of the BG solution was found to be about 6.1 for the concentration range studied and does not change much with the dilution. At a lower *p*H, BG decolorizes due to the structural changes [12], whereas at higher *p*H values, the BG solution becomes unstable and turbid. A similar observation was also noted by many authors[1,3,5,12]. BG is stable at natural *p*H [4],and thus, all the adsorption testswere performed without any *p*H adjustment.

Effect of initial concentration

The process of adsorption is highly affected by the initial solutionconcentration, as an initial concentration provides the necessary driving force to the dye molecule. The initial concentration of dye ranged between 20 and 150 mg/L was studied. The maximum adsorbent dose of SB was maintained at 0.9 g/50 mL at 30°C. Figure 4 shows the change in percentage removal with the change in initial concentration. The percentage adsorption of BG for SBreduced from 94.41 to 86.46% duringan initial concentration of 20-150 mg/L. It was also found that; the amount of BG adsorbed per unit mass of SB was higher with higher initial concentration (Fig. 4).

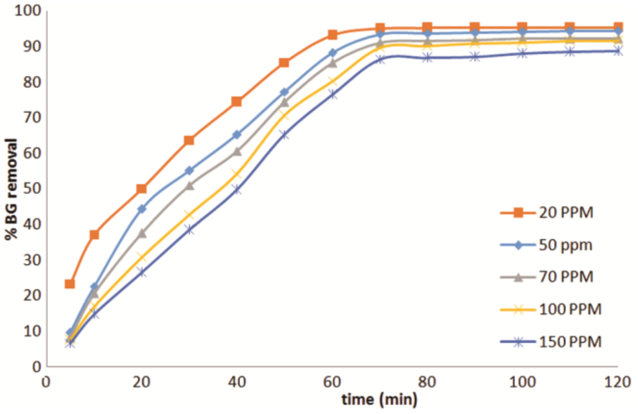


Fig. 3 — Effect of contact time on BG removal onto SB (C_0: 50mg/L, Temperature: 30°C; adsorbent dose: 0.9g/50 mL; rpm: 150)

Effect of temperature

Adsorption study was carried out at temperatures of 30, 40, and 50°C at different initial concentrations between 20 and 150 mg/L at optimal experimental conditions and natural pH of BG. Variation of equilibrium concentration (C_e) and adsorption capacity (q_e) is plotted in Fig. 5. The capacity of adsorption of SB increases with increase in temperature. This suggests the favorability of BG adsorption at a higher temperature on SB.

Adsorption kinetics

The uptake rate of BG on SB can be studied via adsorption kinetics. This removal rate of BG

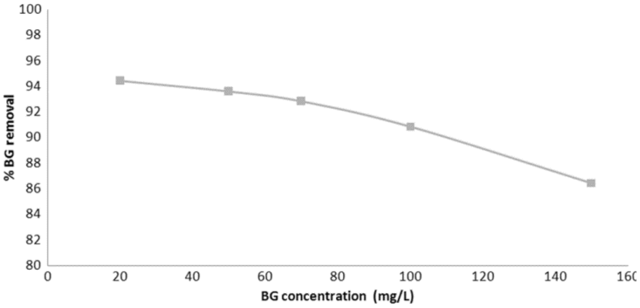


Fig. 4 — Effect of initial concentration on BG removal onto SB (C_0: 50mg/L, temperature: 30°C; adsorbent dose: 0.9g/50 mL; contact time: 70 min; rpm: 150)

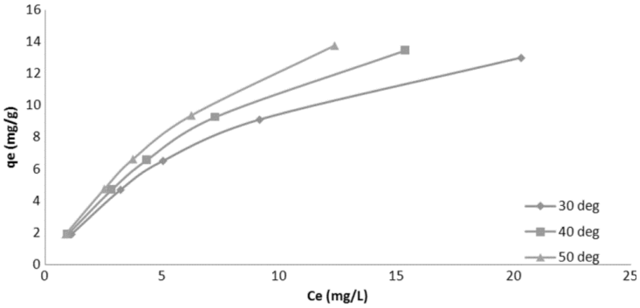


Fig. 5 — Effect of operating temperature on the adsorption capacity of BG onto SB

substantially controls the diffusion process. The adsorption kinetics is governed by many mechanisms, i.e. chemisorption, film diffusion and intraparticle diffusion[13]. During the present study, the kinetic models, i.e. intraparticle diffusion and elovich, pseudo-second-order, Lagergren first-order were explored to understand and explore the adsorption kinetics of BG on SB. The kinetic model used in this study is given in Table 3.

In the intraparticle diffusion model of Weber–Morris, if a straight-line graph of q_t v/s $t^{0.5}$ is observed, then the process of adsorption is predominantly due to intraparticle diffusion only. If plots are multilinear in nature, then it can be deduced that; there are involvement of two or more steps during the process of adsorption. The kinetic parameters calculated for the first-order, second-order, and Elovichkinetic models are listed in Table 4. The R^2 values for the pseudo-second-order model for SB are close to 1, suggesting that the adsorption of BG on to SB follows this kinetic model. The adsorption data are also well fitted to the second-order kinetic model, which highlights that the rate of adsorption of BG is more reliant on the presence of adsorption sites than BG concentration in the solution[15]. The Elovich kinetic model presents good data fit the experimental values (Table 4), shows that that chemisorption, which generally happens by ion exchange, which can be the rate-determining steps during adsorption of BG onto SB[16].

The Weber–Morris plot (Fig. 6) is multilinear in nature, signifying the association of more than one step in the adsorption of BG onto SB. The first section in the Webber–Morris plot represents the resistance to the external mass transfer adjacent to the the adsorbent particle, which is substantial during the early adsorption stages. The process of intraparticle diffusion governs the second portion of the Webber–Morris plot; however, even this stage is characterized by slow adsorption. The third stage represents micropore diffusion. In this stage, the rate of the intraparticle diffusion slows down. This is also called the “equilibrium stage” where maximum adsorption occurs.

Table 3 — Kinetic, isothermal and thermodynamic models used [5,14]

Models	Equation	Graph
Kinetic models		
Lagergren-first-order	$\ln(q_e - q_t) = \ln q_e - k_1 t$	log (qe – qt) versus t
Pseudo-second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_2^2} + \frac{t}{q_2}$	t/q_t versus t
Elovich	$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t$	q_t versus t
Weber–Morris intraparticle diffusion	$q_t = \text{kid}\, t^{0.5} + C$	q_t versus $t^{0.5}$
	Isotherm models	
Langmuir isotherm	$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L}$	$C_e$versus C_e/q_e
Separation factor	$R_L = \frac{1}{1 + C_0 K_L}$	-
Freundlich isotherm	$\log q_e = log k_f + \frac{1}{n}\log C_e$	log q_e versus log C_e
	Thermodynamic model	
van’t Hoff plot	$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$	$\ln k_d$vs. 1/T

Table 4 — Kinetic parameters for adsorption of BG onto SB

C_0 (mg/L)	Lagergren-first-order kinetic model			Pseudo-second-order kinetic model			Elovich model		
	K_1	q_e	R^2	K_2	q_e	R^2	α	β	R^2
20	1.504	1.436	0.882	0.025	1.461	0.978	0.120	3.257	0.965
50	1.407	3.401	0.921	0.003	4.975	0.977	0.198	1.165	0.973
70	1.329	4.777	0.916	0.002	8.130	0.965	0.251	0.853	0.957
100	1.504	6.468	0.931	0.0003	19.604	0.824	0.313	0.631	0.912
150	1.406	9.393	0.919	0.00004	52.631	0.540	0.423	0.444	0.901

In Fig. 6, no line passes through the origin. This gives the nonzero values of Y intercept (*C*) and represents boundary-layer resistance in the early stages of adsorption. The values of *C* (Table 5) represent the thickness of boundary layer, and these increases along with the time, which confirms the increase in the thickness and boundary layer effect on the adsorption of BG on these adsorbents. This observation suggests that during adsorption intraparticle diffusion is involved in; however, this is not the single rate-controlling step[17]. The values of all the constants for the Webber–Morris equation are given in Table 4.

Adsorption isotherm

During the study, Langmuir, as well as Freundlich isotherm, was used to describe the equilibrium adsorption of BG onto SB as shown in Table 3. The equilibrium isotherm has an important role during adsorption system design even the interactions between the adsorbent and solute can also be explained by the adsorption isotherms. The Langmuir isotherm model assumes monolayer adsorption on the adsorbent surface with a fixed number of localized sites without steric hindrance or lateral interactions between the adjacent sites and adsorbed molecules. In Langmuir isotherm, R_L value shows the isotherm type i.e. unfavorable ($R_L > 1$), favorable ($0 < R_L < 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$). The lower value of R_L shows more adsorption favorability[17,18].

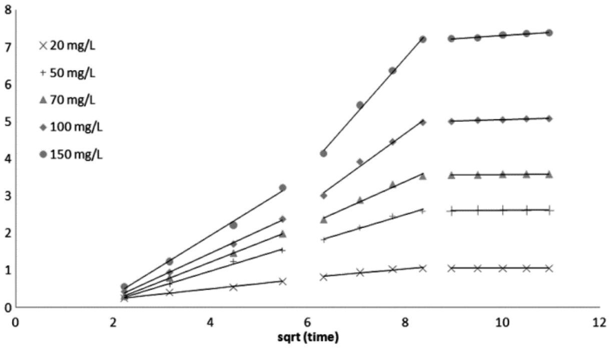


Fig. 6 — Webber Morris plot

The Freundlich adsorption isotherm shows multilayer adsorption and uneven distribution of affinities and heat for adsorption process on the heterogeneous surface. The Freundlich constant (n) shows the adsorption process favorability. The intensity of adsorption is more, or the adsorbent surface is heterogeneous if the slope (1/n) is between 0 and 1. If the slope (1/n) is close to zero, then it shows the increased heterogeneity of the adsorbent surface. The K_F value is adsorbent adsorption capacity for Freundlich isotherm [mg/g $(L/mg)^{1/n}$][17].

The calculated Freundlich and Langmuir adsorption isotherms parameters are givenin Table 6. The Langmuir isotherm results show that it is best fits during adsorption of BG onto SB. This also reveals that the adsorption of BG onto SB is multilayer in nature. The values of $R_L$also confirm that adsorption of BG on SBis favorable under present situations. The maximum Langmuir adsorption capacity for BG removal on SB was 26.32 mg/g. The values of 1/n lie in the range of 1 and 0, showing satisfactory adsorption during all temperatures ranges.

Thermodynamics study

The temperature range of 30-50°C at optimized conditions was studied to explore the effect of temperature during adsorption of BG onto SB.The Gibbs free energy (ΔG)variation is related to the entropy change (ΔS) and Heat of adsorption (ΔH) at constant temperature (Table 4)[17].The ΔH value provides information about the strength of

Table 5 — Webber–Morris constants

C_0 (mg/L)	K_{id1}	C	R^2	K_{id2}	C	R^2	K_{id2}	C	R^2
20	0.134	-0.301	0.994	0.115	0.118	0.939	-0.0003	1.058	0.096
50	0.399	-0.617	0.994	0.392	-0.640	0.984	0.0085	2.521	0.985
70	0.602	-0.944	0.998	0.588	-1.310	0.982	0.0136	3.444	0.832
100	0.513	-0.824	0.999	0.950	-2.917	0.988	0.0381	4.675	0.957
150	0.808	-1.296	0.995	1.486	-5.167	0.995	0.0869	6.447	0.946

Table 6 — Adsorption isotherm parameters for adsorption of BG on SB

T(°C)	Langmuir				Freundlich		
	q_m(mg/g)	K_L(L/mg)	R^2	R_L	K_f(L/g)	N	R^2
30	19.608	0.097	0.999	0.171	1.978	1.504	0.976
40	23.256	0.093	0.999	0.178	2.138	1.407	0.986
50	26.316	0.089	0.999	0.183	2.269	1.329	0.990

Table 7 — Thermodynamic parameters of BG adsorption onto SB

C_0 (mg/L)	ΔH (J/mol)	ΔS (J/mol K)	ΔG (J/mol)		
			30°C	40°C	50°C
20	161.62	28.91	-7123.17	-7796.83	-8286.47
50	157.55	27.45	-6746.43	-7310.84	-7856.56
70	192.21	27.61	-6446.70	-7078.99	-7715.46
100	257.32	27.65	-5783.48	-6631.43	-7276.38
150	344.03	26.81	-4669.18	-5647.46	-6472.79

bonding between BG and SB. Table 7 shows the thermodynamic parameters for BG adsorption onto SB. The value of ΔS which was positive, highlights increased randomness at solid-solution interface as well as the degree of freedom of BG augmentation. Furthermore; the adsorption nature is observed to be endothermic in nature due to positive values of ΔH.

The ΔG value of adsorption of BG onto SB was negative in nature, which is due to the spontaneous nature of the process. The increase in temperature and subsequent decrease in values of ΔG highlights that the adsorption process is favorable during higher temperatures. At higher temperatures, the adsorbent surface is activated and/or there is enlargement of pores on the adsorbent surface. This might be the possible reason for higher adsorption capacity at high temperatures.

The adsorption capacity for BG removal on SB is higher than other reported natural adsorbents; however, the adsorption capacity of SB is less than that of the chemically modified adsorbents. Besides, SB is cheap and easily available industrial waste, and therefore, their use for the removal of BG is economically advantageous.

Conclusion

The present study highlights that, for the effective removal of BG dye from aqueous solutions the utilization of SB as an adsorbent is very environmentally friendly. The removal of BG dye is affected by various parameters, i.e. operating temperature, initial BG concentration, adsorbent dose and contact time. The adsorption of BG onto SB reach equilibrium within 70 min. The adsorption process follows a pseudo-second-order kinetic model, suggesting that the adsorption rate for BG is highly reliant onon the available adsorption sites rather than concentration BG in the solution. The Langmuir adsorption isotherm observed to be better fit during the adsorption process of BG onto SB with the highest capacity adsorption, i.e. 26.316 mg/g at 50°C for SB. At all operating temperatures, the ΔG values were negative which confirms that the BG adsorption was thermodynamically promising and spontaneous in nature. Furthermore; the ΔS positive value highlights the increase in randomness at the interface of adsorbate-adsorbent. Therefore, from results, it can be argued that SB can be used as low-cost and environmentally friendly industrial waste from agriculture material for effective BG dye removal from aqueous solutions.

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