

Removal of Methylene Blue Dye using Carbon Derived from Bulb of *Zephyranthes citrina*: Adsorption and Kinetic Studies

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The studies open up an innovative approach and investigate porous, efficient raw carbon from *Zephyranthes citrina* bulb, which was used as an adsorbent to remove organic dyes. The well-dried and finely powdered *Zephyranthes citrina* bulb was carbonized at 900 °C. The carbonized crude *Z. citrina* sample was characterized by FT-IR, UV-visible, scanning electron microscopy (SEM), BET, X-ray diffraction (XRD) techniques and their adsorption potential to remove the basic methylene blue dye from an aqueous sample. Adsorption studies comprise both adsorption isotherm and kinetic methods. The processes were carried out with diverse adsorbate concentrations and adsorbent quantities at various time intervals in the batch process. Kinetic models of Lagergren first order, pseudo-second order and intra particle diffusion were used to assess the kinetics and adsorption mechanism. The results revealed that the adsorption process follows the first order kinetic model of Lagergren. The BET isotherm model confirmed that it has an excellent adsorption capacity in an adsorption process. Based on the results obtained, the maximum removal (81%) of dye was achieved in a solution containing 50 mg of 50 mL dye at 3 h for methylene blue. The results indicated that the bulb of *Zephyranthes citrina* carbon is a proficient adsorption material and is also used as a cost effective alternative that can adsorb dye from an aqueous solution without activation treatment.

Keywords: *Zephyranthes citrina*, Adsorption isotherms, Kinetics, Methylene blue, Intraparticle diffusion.

INTRODUCTION

Pollution has always been a non-negligible crisis that hampers not only the industrialization process but also the health of people. For example, massive quantities of pollutants are liquefied and hooked on the environment without pretreatment. These pollutants predominantly consists of organic dyes, heavy metal ions, overused antibiotics, different tricky chemicals, etc. [1,2].

The treatment of wastewater from dyeing and finishing processes in the textile industry is one of the imperative environmental problems. Since most of the synthetic dyes have complex aromatic molecular structures, they are inactive and intricate to biodegrade when detached in an environment. Coloured wastes are harmful to aquatic organisms in rivers, lakes and seas, where it is disposed of. In addition, the dyes themselves are an extremely toxic to some organisms and therefore disrupt the ecosystem. Dyes may be the source for allergic dermatitis,

skin irritation, cancer, mutations, etc. Besides, biodegradation of number of dyes produces aromatic amines that are intensely carcinogenic. The continuing exposure of workers in the textile industries are associated with an augmented risk of bladder cancer. Dyes are normally stable to light degradation, biodegradation and oxidizing agents, which has led to intensive research on physical or chemical methods of removing the colour of textile wastewater [3-5]. The effluent treatment studies comprise the use of coagulants, ultrafiltration and electrochemicals. With these methods it has been discovered that adsorption is an efficient and inexpensive process to remove dyes, pigments and other dyes as well as to control the biochemical oxygen demand [6-10].

In recent decades, adsorption, in addition to further purification techniques, has been shown to be a well-established and cost-effective pollutant removal process. The removal of toxic substances, hazardous ions and dyes from industrial wastewater by adsorption is of vast significance for the safety of the

environment and human health. Adsorption by solids reduces the toxicity of the wastewater or removes hazardous organic matter from industrial effluents, etc. [11,12]. Among the accessible adsorbents, the adsorbent derived from plant material is one of the most trendy for both liquid and gaseous purifications because of its unique properties, which consist of its porous structure, highly specific surfaces and large sorption capabilities. The texture and surface properties of carbon are inclined by both the ancestor material and the method used to make it. Conversely, the adsorption efficiency of carbon strapping depends on its specific surface area, the pore size distribution, the surface functional groups present and the later influence its performance through polar interaction with polar, non-polar, anionic and cationic adsorbates [13,14].

Methylene blue, a cationic dye, is the nearly everyone generally used dye for colouring amid all other dyes. Methylene blue may grounds eye burns and if swallowed, irritation of the gastrointestinal tract with symptoms of nausea, vomiting and diarrhea. It can also cause methemoglobinemia, cyanosis, seizures and respiratory distress when inhaled [15]. In this present work, carbon was derived from the bulb of *Zephyranthes citrina* and used as an adsorbent for removal of dye from wastewater samples. It belongs to the family Amaryllidaceae. Plants of this family is a small group of monocotyledonous species, which comprise about 860 to 1100 species in 85 genera distributed largely over tropical and sub-tropical regions. It is a globular plant with green leaves deadlly 4 mm wide. The flowers were lemon yellow colour with cone shaped from 3.1 to 5 cm green tube. The flowers of this rain lily spring forth in late summer. It grows splendidly in natural grasslands and as well as in precincts after rain fall. Since they often come into bloom after it rains, *Zephyranthes citrina* is commonly called as citron zephyr lily or yellow rain lily. To the best of our knowledge, awareness and thoughtful, there is no statement on the consumption of carbon derived from bulb of *Zephyranthes citrina* for the removal of dyes. Hence, the main goal of the current study is an effort for the first time to evaluate adsorption efficiency of carbon from the selected plant in the study of removal of dye by absorption method.

EXPERIMENTAL

Collection and authentication of plant material: The plant material was collected from the crowded area of Institution, Paramathi velur, Namakkal District of India. The plant was renowned and authenticated (Voucher No. BSI/SRC/5/23/2018/Tech/1113) in the BSI (Botanical Survey of India), Department of Botany, Agricultural University, Coimbatore, India. The globular plant objects were cut into pieces, dried under shade for 15 days, coarsely powdered and stored.

Preparation of carbon: *Zephyranthes citrina* was carbonized by using tubular furnace with inert atmosphere at 900 °C for 6 h. After cooling down to the room temperature, the biochar was grained as fine particles. As a final point, the samples were stored in an airtight container for future study.

Experimental methods: Methylene blue was obtained from S.D. Fine Chemicals, Mumbai, India and used as itself devoid of any additional sanitization. Further all the chemicals were used as to A.R. quality. A solution of 1000 mg/L

methylene blue was prepared by dissolving the dye in a double distilled water. Using the above solution, an assortment of concentrations of the solutions was primed and stored in brown glass bottles in order to prevent deterioration from light. Absorbance measurements were made for methylene blue using a UV-visible spectrophotometer. During absorbance measurement, the highest absorption at 665 nm for methylene blue was used for monitoring wavelength. Calibration charts were prepared for methylene blue and concentrations were anticipated using calibration charts.

Characterization: Crystalline structure and phase recognition of the synthesized taster were studied by X-ray diffraction (XRD) techniques (Rigaku-IV Ultima). The morphology and structure were examined by field emission scanning electron microscope (FE-SEM) (FEI Quanta-250 FEG microscope). The optical studies were carried out by using UV-visible spectrophotometer (Jasco V-650).

Batch adsorption experiments: Adsorption experiments for methylene blue dye were performed to examine the consequence of different characters such as initial adsorbate concentration, contact time, adsorbent dose and initial pH. Solutions consists of the preferred concentrations of methylene blue to naturally pH 6.0 and 50 mg g⁻¹. Adsorbent was placed in conical flask (100 mL) and enthused at 200 rpm and 35 °C in a WiseCube shaking incubator. Following the encoded intervals of time, the samples were removed and the supernatant alienated from the adsorbents by centrifugation at 2500 rpm for 20 min. Then the concentration of lingering dye was dogged as indicated above. The consequence of pH was in the range of 2.0 to 10.0 by adjusting the pH of solutions using 1 M HCl and/or 1M NaOH solutions using a pH meter.

Desorption studies: The addition of 10 mg L⁻¹ methylene blue dye in a solution was done by using the adsorbent *via* centrifugation. Using Whatmann filter paper, dye-loaded adsorbent was alienated and tenderly washed with water to eradicate unadsorbed dye. Numerous samples of this type were equipped. Afterward, the spent adsorbent was stirred with 50 mL of double distilled water and then washed with ethyl alcohol. The desorbed dye was anticipated as earlier.

RESULTS AND DISCUSSION

XRD analysis: Fig. 1 shows the XRD spectrum of raw carbon material. It shows a idiosyncratic asymmetric broad peak, which is at about $2\theta = 25^\circ$ and accredited to the reflection (002) of graphite, even though it is enormously disperse in comparison to the ideal graphite, whereas the spiky peak at $2\theta = 44^\circ$ corresponds to the reflection of the crystal phase of (100) graphite. Feeble diffraction peaks at $2\theta = 51^\circ$ associated with the plane (004). There are sharp and wide peaks which are due to the better alignment of the layer and the amorphous structure in the carbon.

FTIR analysis: By using FTIR spectroscopy, the natural environment of the functional groups of the adsorbents and chemical bond, without loaded and with methylene blue loaded were monitored. FTIR spectra were noted in KBr. Fig. 2a shows that FT-IR spectrum of *Zephyranthes citrina* has feeble and wide peaks in the range of 4000-400 cm⁻¹. The FT-IR spectra of carbon indicated that the bands at 3414.07, 2917.88,

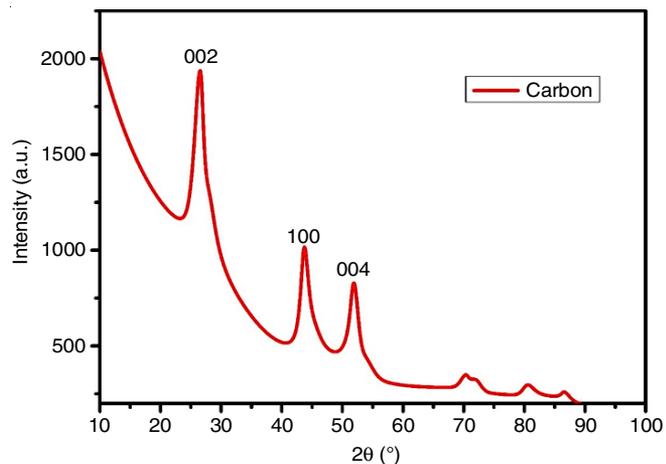


Fig. 1. XRD pattern for carbon of *Zephyranthes citrina*

2851.02, 1552.49, 1035.95, 604.10 cm^{-1} corresponds to -OH, -CH₂, C=O, C=C, CO-C, C-OH (twist broad). These outcome of the results recommend that the presence of oxygen-containing groups, like -COOH and -OH, in *Zephyranthes*

citrina carbon, which probably plays a vital role in methylene blue dye adsorption owing to the electrostatic interactions.

UV and photoluminescence analysis: The UV absorption spectra of *Zephyranthes citrina* carbon was recorded using the UV visible spectrophotometer in the wavelength range about 200-400 nm as shown in Fig. 2b. The peak at 277 nm ascribed to π - π^* transition of the carbon. Furthermore, Fig. 2c shows a detailed photoluminescence investigation with two diverse excitation wavelengths. The photoluminescence spectra of *Zephyranthes citrina* carbon obtained in the range of 300-500 nm at room temperature under excitation wavelength of 270 and 290 nm. The maximum emission peaks was obtained in the range of 360-370 nm. No more significant changes in photoluminescence studies and the visible light emission were obtained because of the absorption (carbon size dependent).

SEM analysis: The scanning electron microscope (SEM) images show a carbon with uniform porosity and surface area. Pores and size of pores increase with increasing carbonization temperature (Fig. 3). Pores and high surface area in the material supports to increase the proficiency of dye molecule. In carbon, dye molecules were absorbed into the pores of the materials.

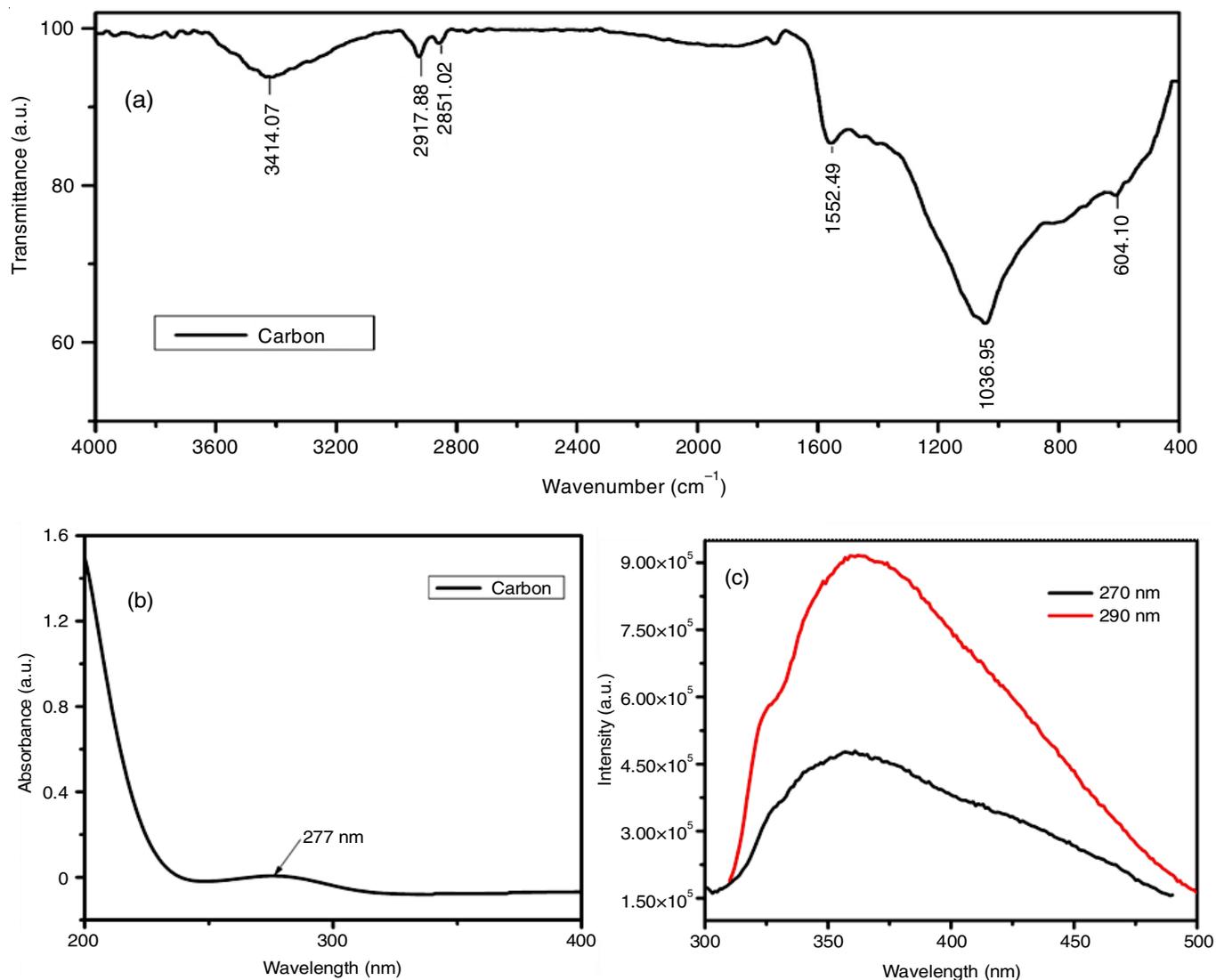


Fig. 2. FT-IR spectra for (a) carbon of *Zephyranthes citrina* and (b) UV spectra of carbon of *Zephyranthes citrina* (c) PL spectra of carbon of *Zephyranthes citrina*

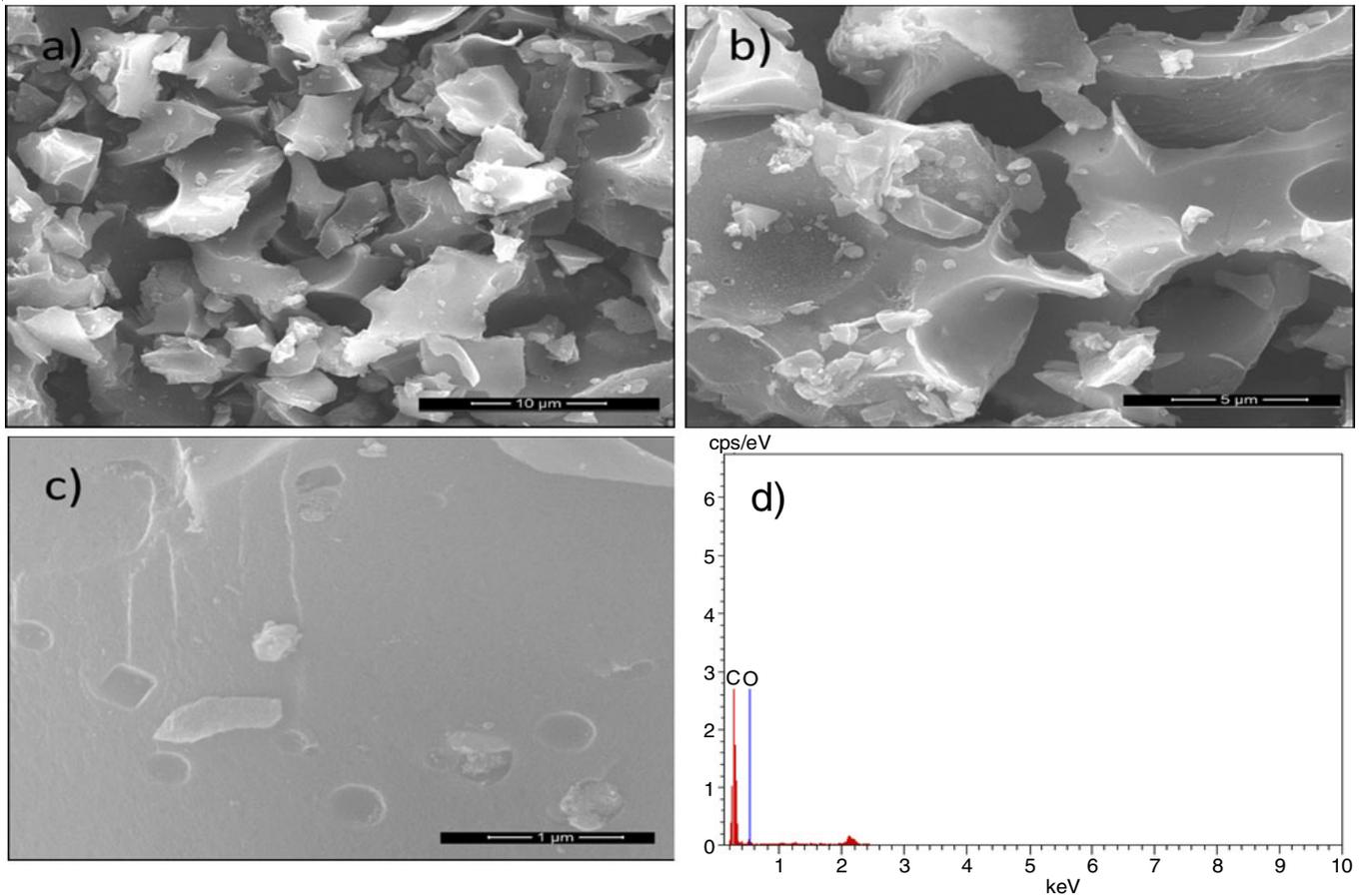


Fig. 3. SEM images of (a-c) *Zephyranthes citrina* carbon (10 μm , 5 μm , 1 μm) and (d) EDAX image of *Zephyranthes citrina* carbon

Fig. 4a indicates the SEM micrographs of carbon with a cubic dye molecules presence on the surface. After adsorption, the dye molecules were present inside the pores and sheltered on the outside of substance. The EDAX spectrum (Fig. 4b) also shows the confirmation of dye molecule present in the surface

and pores of carbon. The efficiency of the sample was increased due to the morphology (porous) of carbon.

BET analysis: The adsorption ability of *Zephyranthes citrina* bulb depends on the size, volume, shape and precise surface area of the pore. The specific surface area of *Z. citrina*

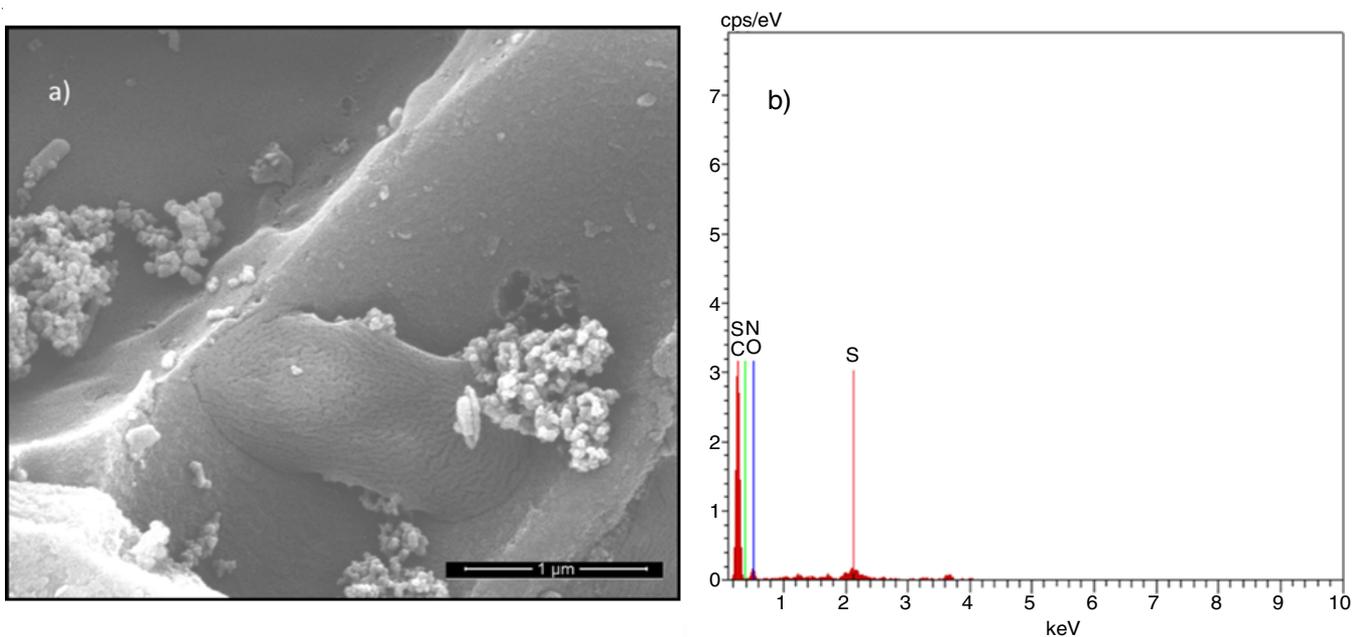


Fig. 4. SEM images of (a) *Zephyranthes citrina* carbon in after adsorption (1 μm) and (b) EDAX image of *Zephyranthes citrina* carbon in after adsorption

carbon was evaluated from BET analysis and the volume of the micropores (V) was determined by calculating the adsorbate volume. In the current study, *Z. citrina* carbon with the largest surface area was prepared by activation at high temperature. The porous structure of *Z. citrina* carbon is based on the fact that the lateral bonds in the molecules are broken and the pore density increases. The S_{BET} , micropore area and average pore diameter of *Zephyranthes citrina* were observed to be $422.16 \text{ m}^2 \text{ g}^{-1}$, $1.93 \text{ m}^2 \text{ g}^{-1}$ and 8.068 \AA , respectively. In support of present findings, IUPAC completed from the results of S_{BET} that the occurrence of a Type I isotherm at high pressure on a horizontal plateau could result in the progress of microporous material dispersed in narrow pores. The results indicated that the modified in the fractal dimension was unrelated and the entire progress of pores on the surface area of *Z. citrina* carbon [16,17].

Batch mode adsorption studies

Effect of pH: The removal of methylene blue based on the effect of pH is shown in Fig. 5. The adsorption efficiency of methylene blue was primarily affected by the surface charge in adsorbent, which in turn is pretentious by pH. The percentage of dye elimination for *Zephyranthes citrina* carbon reduced from 92 to 48% as the pH range of diverse concentrations of dye (10 to 50 mg/L) increased from 2.0-10.0. The decrease in the pH of methylene blue dye solution caused an equivalent increase in adsorption efficiency. At minimum pH, two SO_3^- groups present in the dye grounds protonation, and consequently the electrostatic force of attraction among the protonated dye and the positive charge on the adsorbent surface leads to an increase in adsorption [18,19].

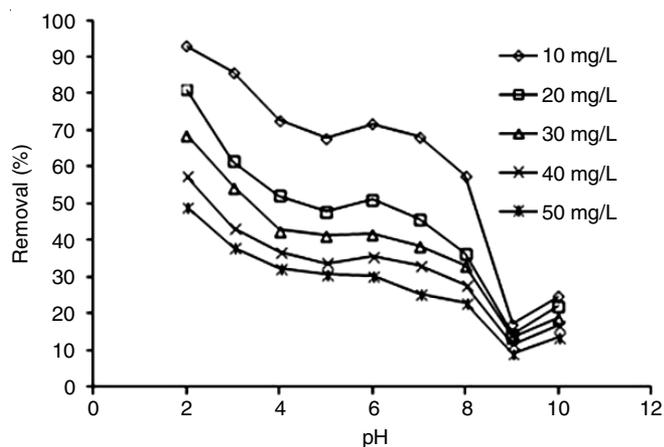


Fig. 5. Effect of initial pH on adsorption of methylene blue onto carbon of *Zephyranthes citrina*

Desorption studies: The influence of pH on desorption of methylene blue on *Zephyranthes citrina* adsorbate-loaded adsorbent at various pH 2.0-10.0 is shown in Fig. 6. The smallest and highest desorption was 8.00 and 43.75% at pH 2.0 and 9.0, respectively for 10 mg/L. The desorption studies have proven that ion exchange mechanisms seem to be the main adsorption type for *Zephyranthes citrina* [20].

Effect of agitation time: A sequence of contact time testing was performed to adsorb methylene blue dye at various primary concentrations (10 to 50 mg/L) at 35°C . The equilibrium time

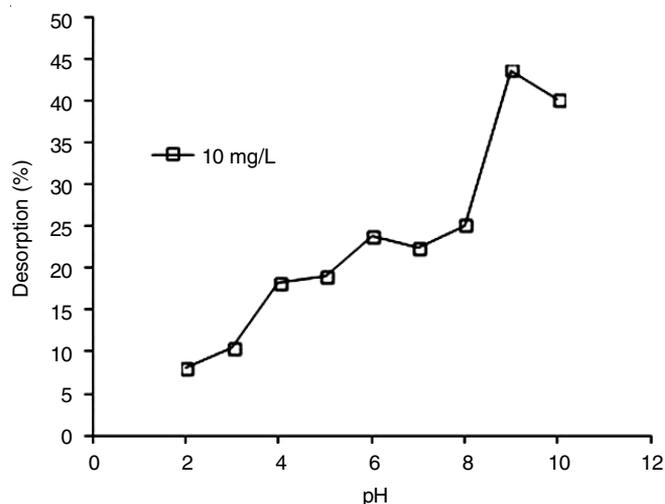


Fig. 6. Effect of initial pH on desorption of methylene blue onto carbon of *Zephyranthes citrina*

for methylene blue was 120 to 10 mg/L, 140 to 20 mg/L and 160 min for the lingering concentrations. The adsorption delayed at a later time because primarily a huge amount of free surface sites were accessible for adsorption and the residual free surface sites may become tricky to engage after a while owing to the revolting forces among the molecules in the adsorbent and bulk phases. In general, initial concentration provides an imperative driving force to overcome the overall mass transfer resistance of dye involving the aqueous and solid phases. Fig. 7 shows the equilibrium adsorption capability of methylene blue at the primary dye concentrations of 9.83 to 38.9 mg/L at 10-50 mg/L [21].

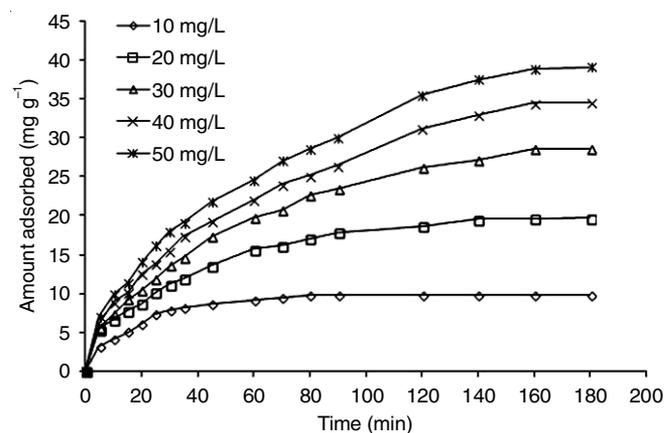


Fig. 7. Effect of agitation time on adsorption of methylene blue onto carbon of *Zephyranthes citrina*

Effect of adsorbent dose: Fig. 8 shows the elimination of methylene blue by *Zephyranthes citrina* carbon at various doses of the adsorbent (10-50 mg/L). It was found that the elimination percentage for *Zephyranthes citrina* was 91.83 to 100%, in doses of 50 to 200 mg. The results in Fig. 8 confirmed that by increasing the dose of an adsorbent, the whole number of adsorption sites becomes increased. Hence, percentage of dye removal increased.

Adsorption kinetics: The Lagergren equation is one of the most commonly used adsorption rate equation for calculating adsorbate adsorption from an aqueous solution. The

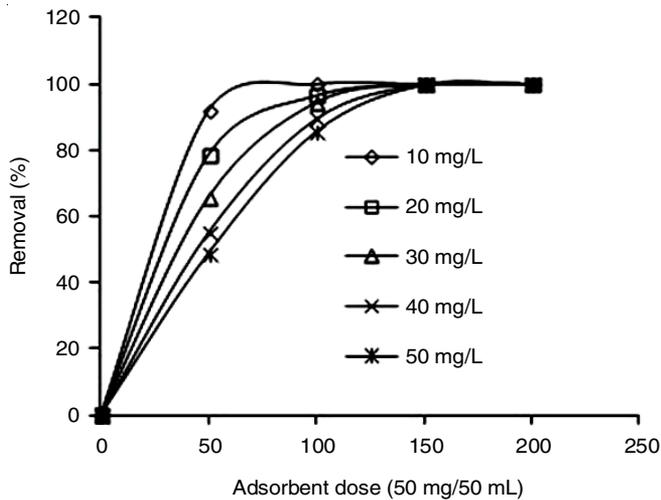


Fig. 8. Effect of adsorbent dose on adsorption of methylene blue onto carbon of *Zephyranthes citrina*

Lagergren of the first-order rate equation may be represented as follows:

$$\log(q_e - q) = \log q_e - \frac{k_1 t}{2.303} \tag{1}$$

where, q_e and q are the amounts of dye adsorbed (mg/g) at equilibrium and at time t (min) and k_1 is the Lagergren rate constant for first-order adsorption (per min).

The pseudo second-order kinetic may be written as

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \tag{2}$$

where, k_2 is the equilibrium rate constant for adsorption of pseudo second-order ($g\ mg^{-1}\ min^{-1}$).

The calculated results from the first and second-order kinetic models together with the experimental q_e values are shown in Table-1. In general, the deliberate q_e values of first-order kinetics of Lagergren were closer to the experimental q_e values, compared with the values of q_e deliberate from the second-order kinetics for methylene blue in *Zephyranthes citrina* carbon. As a result, adsorption follows the first-order Lagergren kinetic model for methylene blue dye [22].

Intraparticle diffusion: The present study also includes the intraparticle diffusion model based on the kinetic data measured and expressed by Weber-Morris equation:

$$q_t = k_{id} t^{1/2} \tag{3}$$

where, q_t is the amount adsorbed at time t , and k_{id} is the intraparticle diffusion rate constant.

The absorption rate could be limited by the size, adsorbate concentration, its affinity for the adsorbent, diffusion coefficient and pore size distribution of the adsorbent. Due to the disparity in size of the inner pores and a different approach for entire sorption period, the mechanism may be changed [23]. Based on eqn 3, the slopes of the linear parts of the graphs of q_t versus $t_{1/2}$ indicate the values of k_{id} as shown in Table-1. As can be seen in Fig. 9, the graphs are non-linear over the entire time domain, which implies that more than one process was pretentious by adsorption. The multiplicity of these graphs could be enlightened by the diffusion of the boundary layer, which gave the first part and the intraparticle diffusion, which

Kinetic model	Conc. (mg/L)	q_e exp (mg/g)	k_1 (min^{-1})	q_e cal (mg/g)	R^2
First order	10	9.83	0.051	10.05	0.978
	20	19.59	0.023	17.22	0.996
	30	28.64	0.018	27.54	0.989
	40	34.54	0.018	34.83	0.958
	50	38.9	0.021	40.74	0.943
Kinetic model	Conc. (mg/L)	q_e exp (mg/g)	k_1 (min^{-1})	q_e cal (mg/g)	R^2
Second order	10	9.83	0.0058	11.36	0.996
	20	19.59	0.0015	23.26	0.988
	30	28.64	0.0006	35.71	0.980
	40	34.54	0.0005	41.67	0.969
	50	38.9	0.0005	47.62	0.966
Kinetic model	Conc. (mg/L)	q_e exp (mg/g)	k_{id} ($mg\ g^{-1}\ h^{-1/2}$)	C	R^2
Intraparticle diffusion	10	9.83	0.566	3.713	0.773
	20	19.59	1.395	3.261	0.949
	30	28.64	2.223	1.229	0.982
	40	34.54	2.697	0.662	0.993
	50	38.9	3.059	0.674	0.992

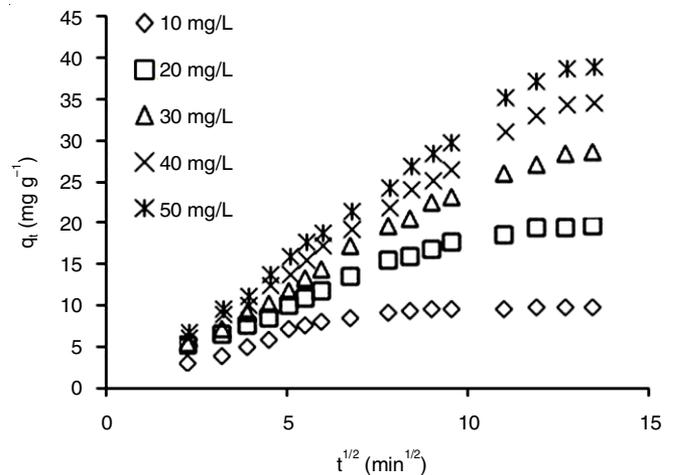


Fig. 9. Intraparticle diffusion plots for adsorption of methylene blue onto carbon of *Zephyranthes citrina*

gave an additional linear part. If intraparticle diffusion was the only speed control step, the plot would pass through the origin, otherwise, diffusion of the boundary layer controlled the adsorption to some degree [24]. This oblique that the dye molecule intraparticle diffusion in the mesopores was the rate restricted step in the adsorption process of *Zephyranthes citrina* carbon, especially during long contact times.

Adsorption isotherms: The adsorption isotherm is significant from a theoretical as well as a practical point of view. So, as to optimize the sketch of an adsorption system for the removal of dyes, it is imperative to determine the most essential correlations of the equilibrium data of each system. The data collected from the various replica offer significant information on sorption mechanisms, surface properties and adsorbent affinities. This equilibrium of adsorption could be articulated by the isotherms of Langmuir, Freundlich and Dubinin-Radushkevitch (D-R) [25].

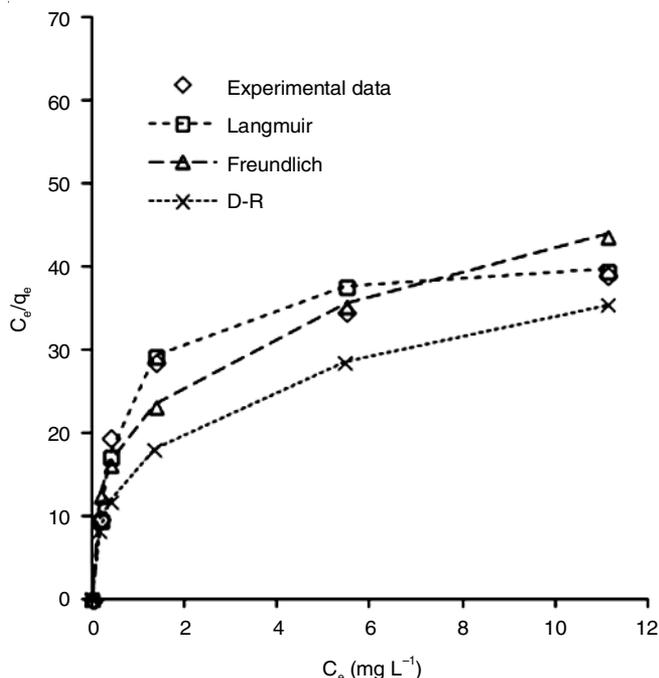


Fig. 10. Comparative assessment of Langmuir, Freundlich and D-R isotherms for adsorption of methylene blue dye onto *Zephyranthes citrina*

brium experimental adsorption data for methylene blue on *Zephyranthes citrina* carbon. The comparison of the adsorption capacity with some other adsorbent is shown in Table-3.

Adsorbent	Langmuir Q_0	Ref.
<i>Mansonia</i> wood sawdust/299 K	17.7	[32]
Sugarcane dust	3.79	[33]
<i>Neem</i> sawdust	3.79	[33]
<i>Pinus</i> bark powder	32.8	[34]
Wood apple	19.8	[34]
Banana pith	5.92	[35]
Wheat Bran	6.410	[36]
Almond shell	11.95	[37]
<i>Aloe vera</i> plant ash	29.81	[38]
<i>Zephyranthes citrina</i> carbon	41.67	Present Study

Conclusion

The adsorption of methylene blue dye from an aqueous solution using carbon derived from the bulb of *Zephyranthes citrina* has been successfully investigated. Batch mode adsorption studies were performed to appraise the effect of diverse structure such as initial concentration of dye, pH, adsorbent dose, agitation time and desorption for methylene blue dye removal by *Zephyranthes citrina* carbon. The chemical properties of both the adsorbate and adsorbent in an aqueous solution would be affected by pH. It was found that the maximum removal of dye for adsorbent was pH 9.0. The results revealed that the adsorption goes behind the first order kinetics of Lagergren. Adsorption equilibrium data were indulged by isotherm equations of Langmuir, Freundlich and Dubinin-Radushkevich (D-R). The experimental data showed an excellent agreement

with the Langmuir isotherm model. With increasing the concentration, desorption of adsorbed methylene blue also increased, suggesting that the adsorption mechanism occurs through ion exchange. Hence, *Zephyranthes citrina* can be used as a low cost material and environmentally benign for the recovery of dyes from an aqueous solution and the possibility of reusing carbon.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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