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Fast adsorptive removal of methylene blue dye from aqueous solution onto a wild carrot flower activated carbon: isotherms and kinetics studies

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ABSTRACT

In the present study, adsorption of methylene blue (MB) dye from aqueous solution on to a wild carrot flower activated carbon (WCFAC) as a potential adsorbent was investigated. No attempt has been made in the past on WCFAC and it also finds application in reducing the population of this poisonous weed. WCFAC was prepared using simple chemical activation method and MB dye adsorption was studied by batch adsorption experiments. The effects of various parameters like dosage effect, effect of pH, initial concentration of the dye and contact time on the adsorption capacity of MB dye on WCFAC are reported. It was found that, maximum adsorption (~88%) was observed at pH 6 with a good sorption capacity (~21 mg/g) of dye. The adsorption isotherms are analyzed by Langmuir and Freundlich models. The sorption data were better fitted by the Freundlich isotherm model as evident from the R^2 value 0.940. Adsorption kinetics was performed and obeys pseudo-second-order kinetic model (R^2 at 0.9325). The zero point charge for the WCFAC was found to be 4.9 pH. pH optimization studies show that the favorable adsorption range ~4–6 pH. Experimental and kinetic results reveal that WCFAC is a potential adsorbent for the adsorption of MB dye from aqueous solution.

Keywords: Wild carrot; Methylene blue; Langmuir; Freundlich; Adsorption

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1. Introduction

With growing advances in science and technology, there was a bigger demand on opting for newer chemicals which could be used in various industrial processes. Among many new chemicals, organic dyes came up as one of the most widely used chemical stuff which could be used in many industrial activities. Number of industries uses dyes to give color to their products and residual unspent dyes are discharged into the rivers and other natural water bodies. This affects particularly aquatic environment [1,2], dyeing in textile industries, introduce organic dyes into the environment via their waste effluent [3]. Even dyes at very low concentrations in effluent are highly visible and are considered undesirable, especially blue color, because of its wavelength range [4,5]. Many azo dyes may decompose under aerobic conditions into potential carcinogenic aromatic amines and these reactive dyes are the most problematic compared with other forms of dyes [6]. The removal of dye from water sources is important in terms of protecting public health and environment [7,8]. Dye induced toxicological and pathological problems like cardiovascular, dermatologic, gastrointestinal,

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genitourinary, hematologic and central nervous system damage. Therefore, removal of these dyes from wastewater is a major environmental challenge and there is a constant need to have an effective process that can efficiently remove these dyes economically [7]. However, the conventional biological treatment process is not very effective in treating dyes from wastewater, due to low biodegradation and bioefficacy [9].

Ion exchange, phytoextraction, ultrafiltration, membrane separation and photocatalysis are the usual methods for removal of dyes from aqueous solutions [10]. These treatment processes are very expensive, time consuming, the process is tedious and could not be effectively used to treat the wide range of complex dyes from wastewaters. However, adsorption process was proved to be an alternative and simple method for the removal of toxic chemicals like the dyes [10]. Adsorption process was extensively used for removal of dye from aquatic medium using low cost adsorbents such as plant materials and cellulose materials [11]. Among them activated carbons prepared by agricultural by-products and biomass are relatively cheap and show very high adsorption capacity, due to simplicity and ease of operational conditions adsorption is a widely used process [12].

Researchers have studied the production of activated carbon from coal, coconut shells, almond shells, olive stones, rice husks, nutshells [13], coconut tree sawdust [14], coir pith [15] and other agricultural products using physical and chemical process [16], it finds applications in the removal of various metal ions and dyes from wastewater [17].

However, the adsorption capacity of activated carbon for a specific adsorbent varies strongly with the type of material and the processing technique used [18]. The feasibility of producing carbon from agricultural wastes depends on a number of factors, including the adsorption capacity and mechanical strength of the raw material and the cost of production [19,20]. Activated carbons prepared from coconut shells.

Wild carrot weed, commonly known as *Parthenium hysterophorus* is one of the seven most dangerous toxic weeds of the world and has assumed the status of a major health hazard [21]. The colonization efficiency of it is higher than any other weed in India. Therefore, neither its containment nor its eradication is practically possible in a long-term prospective without the total involvement of the government and the public, and it has no known beneficial use [22]. Some previous studies found in the literature for removal of methylene blue (MB) by using *P. hysterophorus* and its activated carbon, but none of the authors gives importance to its flowers. No study has been reported in *Parthenium* flower activated carbon. The biomass of *Parthenium* flowers is not put to any use and increasing its population and harmful effects [9]. The focus of our present research work is on the adsorptive removal of MB dye onto a wild carrot flower activated carbon (WCFAC). To investigate the adsorption efficiency by batch adsorption experiments study kinetics under various conditions like pH, contact time, dosage of adsorbent and concentration of dye.

2. Materials and methods

The chemicals used in this study are pure sulphuric acid (H_2SO_4, AR 98%, Sigma-Aldrich, Bangalore, India), hydrochloric acid (HCl, AR 99%, Sigma-Aldrich, Bangalore, India), sodium bicarbonate ($NaHCO_3$, AR 99%, Sigma-Aldrich, Bangalore, India), methylene blue ($C_{16}H_{18}N_3SCl$, AR 85% Merck, Bangalore, India). All the chemicals were used as such without further purification. Double distilled water was used to prepare the solutions.

2.1. Preparation of WCFAC adsorbent

The creamy-white color flowers from fully grown wild carrot plants were collected in and around the local areas of the Kerehalli village of Chamarajanagara district in Karnataka state of India. The flowers are dried in hot air oven for 2–3 d and ground into powder and used for the preparation of activated carbon biomass by fully soaking with concentrated H_2SO_4, keeping it at 200°C for 24 h. The carbonized material was then washed with $NaHCO_3$ solution for several times to remove the acidic contents and further soaked in 0.1 N $NaHCO_3$ solution for 6 h. The resulting product was washed with double distilled water, dried at 110°C in a hot air oven for 24 h and it was ground well to get fine powder of WCFAC.

2.2. Preparation of adsorbate

The accurately weighed 25 mg of MB dye was dissolved in 1,000 mL of double distilled water to prepare the stock solution (25 mg/L). The percentage purity of the dye solution was taken into consideration while preparing the stock solutions. Experimental solution of desired concentration was obtained by successive dilution.

2.3. pH at point of zero charge

The pH_{PZC} (point of zero charge) is pH when the charge on the surface is zero. The procedure of pH drift method [23] could be described as follows: to a series of 250 mL conical flasks, 50 mL of 0.01 N NaCl was added. Then, the initial solution pH values (initial pH) were adjusted in range between 2 and 12 of using 0.01 N HCl solutions and 0.01 N NaOH. After constant value of initial pH had been reached, 0.010 g of WCFAC sample was added into each conical flask and caped them immediately. This mixture was kept undisturbed for 12 h till reaching equilibrium. After 12 h, pH of solution was measured and noted as pH final. The pH_{PZC} of WCFAC sample is the point when pH initial = pH final.

2.4. Batch adsorption studies

Batch experiments were carried out at different contact time, dose, pH and initial concentration of dye. 100 mL of dye solution of desired concentration of 5 mg per liter was mixed with different doses of adsorbent in 250 mL beaker at lab temperature and different contact time. The dye solution is separated from the adsorbent by centrifugation at 1,200 rpm for 5 min. Residual concentration of dye in the supernatant was estimated spectrophotometrically by monitoring the absorbance at 664 nm (λ_{max}) using a UV–Vis spectrophotometer (Model ELICO SL 159).

3. Results and discussion

3.1. Effect of adsorbent dose

The effect of adsorbent dose on the percentage adsorption at equilibrium conditions is shown in Fig. 1. It was observed that the amount of MB adsorption increases with the increase in the adsorbent dosage. As the adsorbent dosage increase from 1 to 5 mg, the percentage adsorption is increased from 82% to 86 %. Above 5 mg of WCFAC, the adsorption became slow because saturation of adsorption sites and dye molecules in solution [24–26].

3.2. Effect of pH

The pH of adsorption medium influencing the surface charges of the adsorbent and thereby plays an important role in adsorption capacity. In addition, the degree of ionization of the material present in the solution and the dissociation of functional groups on the active sites of the adsorbent also affects the adsorption [27]. The effect of solution pH on the amount of MB adsorbed was studied by varying the pH under constant process parameters at equilibrium conditions. Batch experiments were performed by varying pH from 2 to 12 at room temperature, the agitation time was 30 min and 5 mg of optimized quantity of WCFAC was used and results are shown in Fig. 2. It is observed that the MB adsorption was strongly dependent on pH. The maximum removal efficiency (88%) of the WCFAC was found at the pH 6 and at higher pH values, the removal efficiency decreases gradually, because MB is basic dye.

3.3. Effect of contact time

The evaluate stirring time, the batch experiments were carried out with 100 mL of 5 mg per liter dye solution at room temperature, by adding optimized quantity of 5 mg of WCFAC the results are shown in Fig. 3. The adsorption of MB onto a WCFAC increases with time until the equilibrium is attained in solution. It was observed that, the uptake of MB was high in the beginning, followed by a much slower subsequent adsorption and at last leading to steady state. About 80% of the MB was adsorbed by the WCFAC in the first 30 min of the reaction, while only a small part of the additional removal occurred in the rest. This rapid adsorption may be attributed to the external surface adsorption. This suggests that most of the adsorption sites of the WCFAC existed in the exterior of the adsorbent and were ease accessible by the MB species resulting in a rapid approach to steady state [28,29].

3.4. Effect of initial dye concentration

The initial concentration of dye is also another parameter which needs to be taken into account. The adsorption of MB was carried out at different initial dye concentrations ranging from 5 to 25 ppm at 30 min contact time using optimized quantity 5 mg of the WCFAC and at pH 6. It is very interesting to note that the percentage of adsorption decreases as dye concentration increases (Fig. 4) because adsorption sites are not enough to adsorb the dye molecules at higher concentration [30]. With decreasing the concentration of dye 25–10 ppm, percentage of adsorption increases, further 10–5 ppm of MB concentration, percentage of adsorption onto the WCFAC was found to be good adsorption. This experimental result clearly explains that availability of dye molecules to interact with the adsorbent should be in the optimum range. This indicates that the initial concentration provided

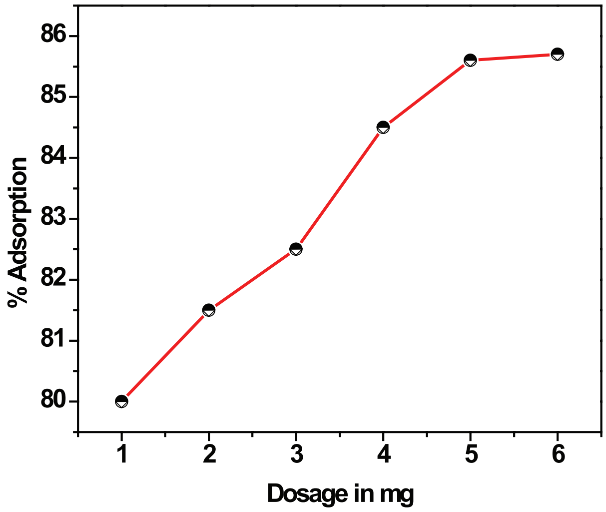

Fig. 1. Effect of adsorbent dosage on adsorption of MB.

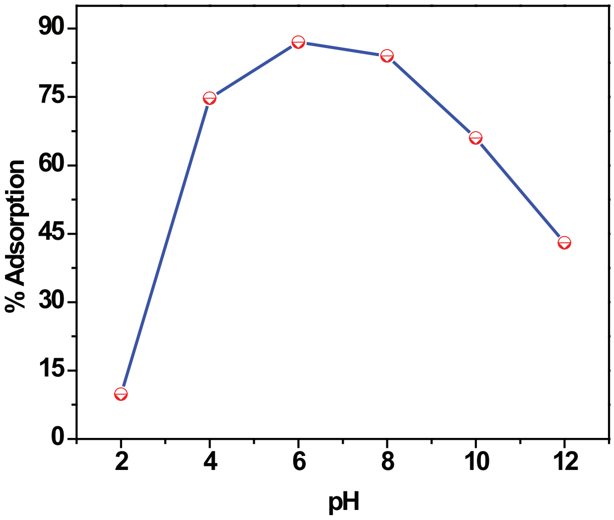

Fig. 2. Effect of pH on adsorption of MB.

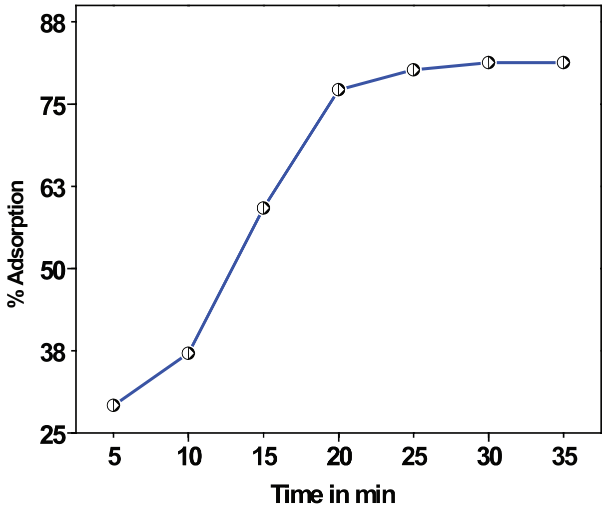

Fig. 3. Effect of contact time on adsorption of MB.

a powerful driving force to overcome the mass transfer resistance between the aqueous and solid phases.

3.5. Desorption studies (regeneration)

Desorption and adsorbent regeneration is a critical step, which contributes to the process costs and pollutant recovery. A successful regeneration process should restore the initial characteristics of the adsorbent, allowing the solid reuse during the maximum number of cycles and thus decreasing the costs of the overall separation process. However, in a large number of sorption studies available in the literature survey with the use of activated carbon and carbon nanoparticles for the removal of heavy metals and dyes, desorption and reuse of the adsorbents has not been accurately analyzed [31]. The selection of a suitable effluent depends on the adsorbate and the adsorbent (desorption property), but other operation variables, such as pH and contact time between the solid and liquid phases and the presence of competitive ions in the solution [32,33]. In desorption study, 5 mg of the WCFAC was added to 100 mL of aqueous MB dye solution, after the adsorption studies, the adsorbent was collected by filtration and washed with deionized water twice to remove any residual dye molecules from the surface of the adsorbent. To this cleaned adsorbent, 100 mL of 0.05 M HCl was added stirred for 30 min after which it was centrifuged at 1,200 rpm for 30 min. The supernatant was analyzed for estimating the concentration of dye molecules. From this analysis, the percentage of dye molecules was calculated and plotted as percentage desorption vs. time in minute as shown in Fig. 5. It shows percentage of desorption decreases like 82 > 79 > 44 > 28 for every recycling process in the order of cycle 1 > cycle 2 > cycle 3 > cycle 4.

3.6. pH at point of zero charge determination

The pH_{PZC} results of the experiments performed with the WCFAC adsorbent, where the pH ranged from 2 to 12. Fig. 6 showed the pH_{PZC} of the WCFAC was 4.9, which indicates the acidic character of the WCFAC surface and below the pH_{PZC} value, the surface of WCFAC is positively charged due to protonation, favoring the adsorption of anions because MB is a basic dye. Above the pH_{PZC}, the WCFAC surface has a negative charge which unfavors the adsorption of anion species [34]. In this respect, Jawad et al. [35] reported that the pH_{PZC} value lies between pH 3.20 and which was attributed to the acid form of sulphuric acid treated activated carbon derived from coconut leaf powder treated with H_2SO_4.

4. Isotherms and kinetic studies

4.1. Langmuir isotherm model

The Langmuir model is applicable for monolayer adsorption the solid surface with a finite number of identical sites, where the sorption of each sorbate molecule on to the surface has equal sorption activation energy. The linear form of Langmuir isotherm equation is given as:

$$\frac{C_e}{q_e} = \frac{1}{K_L \times q_{\max}} + \frac{C_e}{q_{\max}} \tag{1}$$

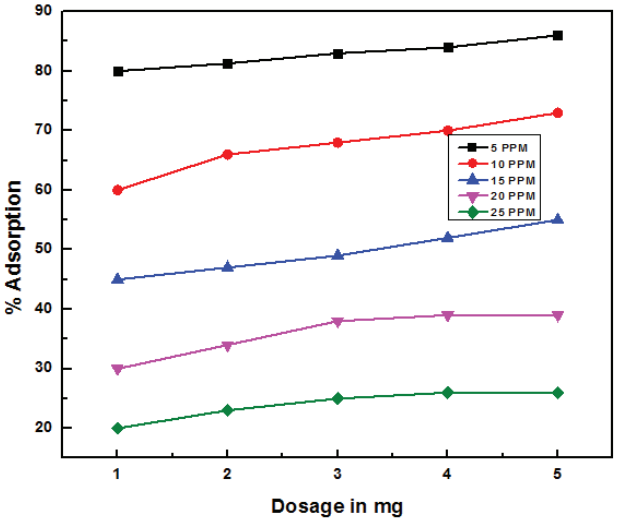

Fig. 4. Effect of initial concentration of dye on WCFAC.

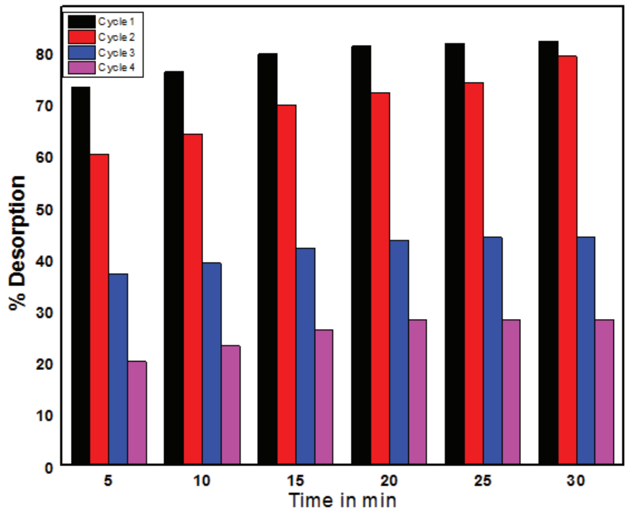

Fig. 5. Desorption (regeneration) studies plot for cycle 1 to cycle 4.

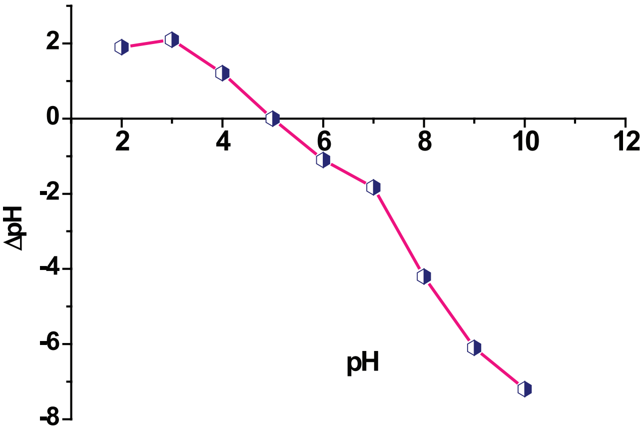

Fig. 6. Zero point charge of WCFAC at 298 K.

where q_e is the amount of MB adsorbed per unit weight of adsorbent (mg/g), C_e the equilibrium concentration of MB in the bulk solution (mg/L), $q_{\max}$ is the monolayer adsorption capacity (mg/g) and K_L is the Langmuir equilibrium constant (L/mg) related to the adsorption. Both are determined from a plot of C_e/q_e vs. C_e (Fig. 7).

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L [36,37], which is defined by the following equation:

$$R_L = \frac{1}{1 + (q_{\max} \times K_L) \times C_0} \tag{2}$$

where C_0 is the highest initial concentration and the value of separation factor, R_L indicates the isotherm type and nature of the adsorption process. Considering the R_L value, adsorption can be unfavorable ($R_L > 0$); linear ($R_L = 1$); favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) [29]. Therefore, as the R_L value closer to zero, the adsorption will be done better. In our experimental study, the R_L value –0.03 (Table 1) unfavorable adsorption, hence non-homogeneous adsorption.

4.2. Freundlich isotherm model

The Freundlich isotherm is purely empirical equation and it best describes adsorption onto a heterogeneous surface [38]. The linear form of the Freundlich equation is given as:

$$\ln q_e = \frac{1}{n} \ln C_e + k_f \tag{3}$$

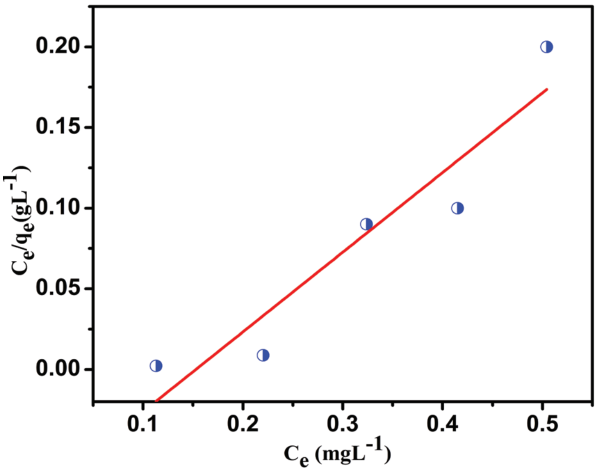

Fig. 7. Langmuir adsorption isotherm for the sorption of MB by WCFAC.

Table 1
Isotherm parameters for adsorption of MB onto WCFAC

Langmuir isotherm			Freundlich isotherm		
q_m (mg/g)	R_L (L/mg)	R^2	K_F (mg/g)	$1/n$	R^2
2.024	–0.03	0.8629	20.859	0.262	0.9352

where k and n are Freundlich constants, with k correlating to the quantity of dye adsorbed on the adsorbent per unit equilibrium concentration and n reflecting how favorable the adsorption process. The value of n greater than unity and R^2 value of the Freundlich plot (Fig. 8) is higher than the Langmuir, suggesting that the equilibrium adsorption of MB onto WCFAC could be best described with the Freundlich isotherm, because the correlation between calculated and experimental values as well as regression factors is in good agreement with Freundlich isotherm indicated that MB was favorably adsorbed by the WCFAC.

4.3. Pseudo-first-order kinetics

In order to find out the potential rate controlling steps involved in the process of adsorption, adsorption kinetics were established. Langmuir–Hinshelwood model expressed in Eq. (4) was applied to understand the adsorption kinetics quantitatively. This model has been used to calculate the rate constant of adsorption experiments [39].

$$r = -dc/dt = k_r KC/(1 + KC) \tag{4}$$

where 'r' is the adsorption rate, 'k_r' is the adsorption rate constant, 'K' is the absorption coefficient of the reactant and 'C' is the reactant concentration [40]. When C is very small, Eq. (4) can be expressed by Eq. (5):

$$r = -\frac{dc}{dt} = k_r KC = kC \tag{5}$$

where 'k' is the first-order rate constant. Set $t = 0$, $C = C_0$, Eq. (6) can be induced.

$$\ln C_0/C = kt \tag{6}$$

Fig. 9 shows the adsorption rate of WCFAC under room temperature. It is clear that the kinetic simulation curve

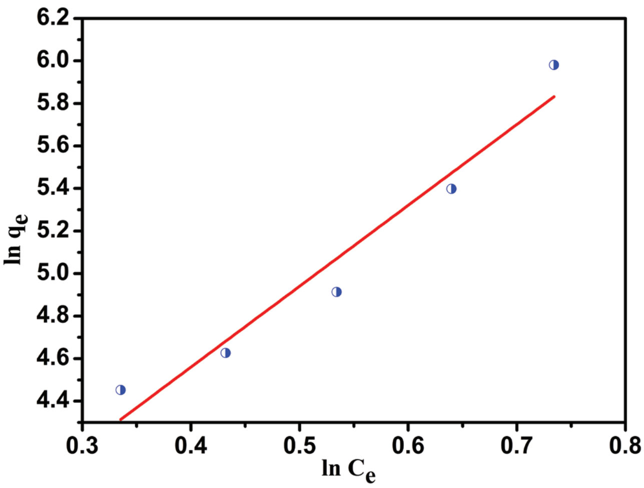

Fig. 8. Freundlich adsorption isotherm for the sorption of MB by WCFAC.

with stirring time (t) as abscissa and $\ln(C_0/C)$ as the vertical ordinate was close to a linear curve with the fitting constant 'R' >0.96. It is observed that WCFAC exhibit good adsorption activity ($k = 0.04$ min^{-1}) [41,42].

4.4. Pseudo-second-order kinetics

Pseudo-second-order kinetics is expressed as follows:

$$\frac{dq_t}{d_t} = k_2(q_e - q_t)^2 \tag{7}$$

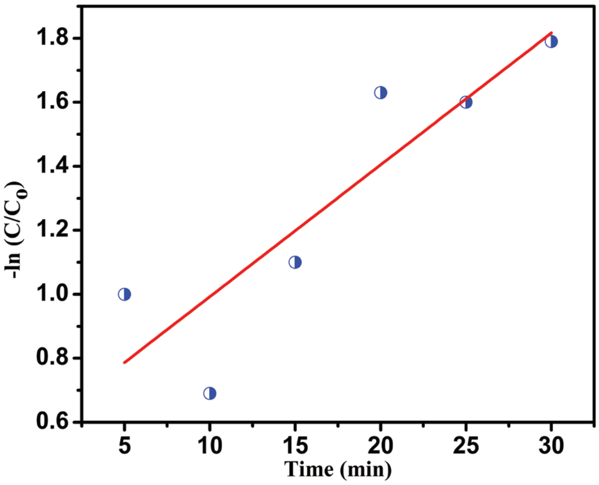

Fig. 9. First-order kinetic plot for the sorption of MB by WCFAC.

Table 2
Kinetics model of adsorption of MB onto WCFAC

First-order kinetics		Pseudo-second-order kinetics		
k_1 (s^{-1})	R^2	q_e (mg/g)	k_2 (s^{-1})	R^2
0.04	0.7364	4.37	0.394	0.9325

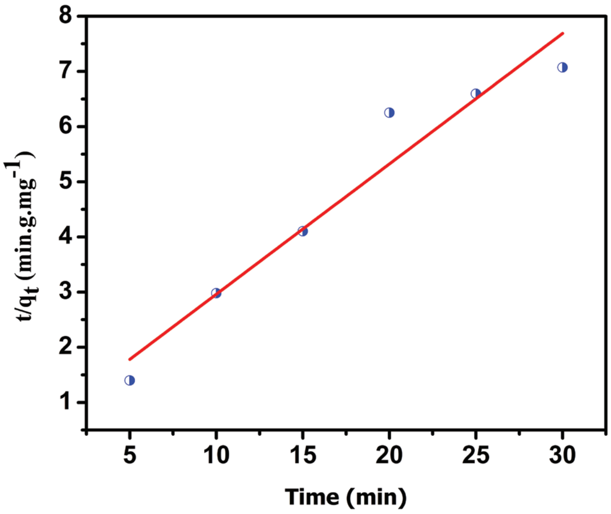

Fig. 10. Pseudo-second-order kinetic plot for the sorption of MB by WCFAC.

where k_2 is the rate constant (g/mg min). Integration of Eq. (7) at the boundary, $q_t = 0$ at $t = 0$ and $q_t = q_t$ at $t = t$ and then rearrangement to a linear form gives Eq. (8).

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{8}$$

The value of k_2 and q_e can be determined from the slope and the intercept of the plot t/q_t vs. t, respectively [43]. As can be seen from Table 2 and Fig. 10, time–concentration data were best fitted to all used kinetic models with higher correlation coefficient onto pseudo-second-order kinetic model [44].

5. Conclusions

This study shows that biowaste, wild carrot flower (*Parthenium* flowers) derived activated carbon can be used as effective low cost adsorbent for removal of a basic dye (methylene blue) from aqueous solution. The adsorption properties under thermodynamic conditions are described by the pseudo-first-order and pseudo-second-order models, while adsorption results at equilibrium are explained by the Langmuir and Freundlich isotherm models. The maximum adsorption capacity (mg/g) of 21 mg/g was obtained at 303 K. The important contribution of this work is that, with significantly less amount of adsorbent loading better color removing efficiency was observed. This study raises the hope for developing an appropriate technology for effective designing of a wastewater treatment plant. However, further work should be performed in order to have a better understanding the parameters for the complete design specifications. It can be concluded that wild carrot flower activated carbon adsorbent has a great potential for removing MB from aqueous solutions as an eco-friendly process.

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