Research Article

Adsorptive Removal of MG Dye from Aqueous Solution on to a PH-FAC: Isotherms and Kinetics Studies

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Abstract

Expelling of dyes into water resource system causes major thread to the environment. Adsorption is the cost effective and potential method to remove the dyes from the effluents. Therefore, an attempt was made to study the adsorption of Malachite Green (MG) dye from aqueous solution by naturally occurring *Parthenium hysterophorous* Flower Activated Carbon (PHFAC) as an excellent adsorbent was investigated. PHFAC was prepared using simple chemical activation method and MG dye adsorption was studied by spectro photometric method. The synthesized material was characterized by Fourier Transform Infrared (FT-IR) spectroscopy before and after adsorption, Scanning Electron Microscopy (SEM). Batch adsorption experiments were used to analyze the effect of various parameters like effect of pH, initial concentration of the dye, adsorbent dose, and contact time on the adsorption capacity of MG dye on PHFAC. It was found that, maximum (95%) adsorption was observed at pH 6 with sorption capacity of 194.6 mg L-1 of dye. The adsorption isotherms were analyzed by Langmuir and Freundlich isotherm models and sorption data was better fitted by the Freundlich isotherm model. Adsorption kinetics also was performed and obeys pseudo second order kinetic model. Experimental and kinetic results reveal that PHFAC is a potential adsorbent for the adsorption of MG dye from aqueous solution.

Keywords: Adsorption; Freundlich; Langmuir; Malachite Green; Pseudo Second Order

Public Interest Statement

My main research has focused primarily on understanding adsorption studies of dyes on plant extract activated carbon materials for efficient applications in wastewater treatment. The field of wastewater treatment has expanded greatly due to the development of high-throughput experimental methodologies, which has significant contribution in solving environmental-related problems that occur by different pollutants, from different sources especially from industries on the water resource. Nowadays, keeping the water quality is a requirement due to climate change, but currently, the demand in using water has been gradually increased in agriculture, industry, and domestic sectors, which consequently produces a large quantity of waste water. Therefore, it needs urgent and effective action in order to maintain the quality of water for the sake of keeping our environment safe.

Introduction

Parthenium hysterophorous (L) also known as wild carrot weed is one of the seven most dangerous toxic weeds of the world and has assumed the status of a major health hazard [1]. 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. It has no known beneficial use [2]. Dyes used in various industries have harmful effects on living organisms within short exposure periods. The disposal of dyes in wastewater is an environmental problem that causes ill effects

to the ecosystem [3]. Dye adsorption is a process of transfer of dye molecules from bulk solution phase to the surface of the Adsorbent [4]. The removal of dyes from water and waste water is important in terms of protecting public health and environment. Many industrial activities such as metal plating industry, fertilizer, mining operations, metallurgy, manufacturing of battery and dyeing in textile industries, introduce organic dyes into environment via their waste effluent [5]. Even dyes at very low concentrations in effluent are highly visible and are consider undesirable, especially the red, blue color [6]. Dye is the organic substance, if reacts with other substances gives variety of colors. The treatment of dye waste water has limited biodegradability in an aerobic environment and many ago dyes may decompose under aerobic conditions into potential carcinogenic aromatic amines [4]. Color is the most obvious indicator of water pollution. These reactive dyes are the most problematic compared to other forms of dyes and must be removed completely [7]. However, removal of these dyes from waste water is a major environmental challenge and there is constant need to have an effective process that can efficiently remove these dyes economically [3].

Ion exchange, Phyto extraction, ultra-filtration, membrane separation and adsorption are the usual methods for removal of dyes from aqueous solutions [8]. Due to simplicity and easy operational conditions adsorption is a widely-used process [9]. Researchers have studied the production of activated carbon from coal, coconut shells, almond shells, olive stones and other agricultural products using physical and chemical process [10]. However, the adsorption capacity of activated carbon for a specific adsorb ate varies strongly with the type of material and the processing technique used [9]. 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 [11]. Adsorption process was extensively used to remove dyes from aquatic medium using low cost adsorbents such as plant materials, cellulose materials. Among them Activated carbons prepared by agricultural byproducts and biomass are relatively cheap and show very high adsorption capacity [12].

Activated carbons prepared from coconut shells, rice husks, nutshells [13], coconut tree saw dust [14] and coir pith [15] find application in the removal various ions and dyes from waste water [16]. Some previous studies found in the literature for removal of MG by using *Parthenium hysteroporous* and its activated carbon, but none of the authors gives importance to its flowers, during that time flowers fell down to the ground and converted into seeds and it increases the population of *Parthenium* weed. No study has been done on *Parthenium* flower activated carbon. In this context, new economical easily available and highly effective adsorbents are still needed. *Parthenium*, also known as carrot weed, an annual herbaceous weed, a native of Mexico as now widely spread in India, China, Australia, Pacific island etc. Parthenium plant contains Parthenon, hymen in and ambrosinect. This may induce allergic dermatitis. It causes several health problems in human beings and animals, e.g. allergy, dermatitis, eczema, black spots, blisters around eyes, burning, rings and blisters over skin, asthma, high fever, etc. The biomass of this plant is not put to any use and disposes along the road sides and agricultural fields, railway tracks after uprooting [8].

The focus of our present research work is on the adsorptive removal of MG dye onto a naturally occurring PHFAC. The batch adsorption experiment and kinetic studies under various conditions like pH, contact time, variation of adsorbent and different dye concentration was carried out and the adsorption mechanism is discussed comprehensively based on the results.

Materials and Methods

Commercially pure Sulphuric Acid (H_2SO_4, AR 98%, Merck, Mumbai, India), Hydrochloric Acid (HCl, AR 99%, Merck, Mumbai, India), Sodium bicarbonate ($NaHCO_3$, AR 99%, Merck, Mumbai, India), Malachite Green (C50H52N4O8; 90% $k$max = 617 nm, S.D. Fine-Chem Mumbai, India).All chemicals were used as such without further purification.

Preparation of PHFAC Adsorbent

Creamy-white colour flowers from fully grown wild carrot plants were collected in and around local areas of Gantiganahalli, Bangalore. The flowers are dried in hot air oven for 2 days and ground into powder and used for the preparation of activated carbon biomass by mixing with concentrated H_2SO_4 and keeping it at 210°C for 24 hours. The carbonized material was then washed with $NaHCO_3$ solution for several times to remove the acidic contents. That product was then washed with double distilled water and dried at 100°C in a hot air oven for 20 hours. It was grind well to get fine powder of PHFAC.

Preparation of Adsorbate

The accurately weighed 5 mg quantity of the MG dye was dissolved in double distilled water to prepare the stock solution (5 mg/L). The percentage purity of the dye was taken into consideration while preparing the stock solutions. Experimental solution of desired concentration was obtained by successive dilution.

Batch Adsorption Studies

Batch experiments were carried out at different time, dose, pH, and initial concentration of dye. 100 ml of dye solution of desired concentration was mixed with different dose of adsorbent in 250 ml conical flask at lab temperature. The dye solution is separated from the adsorbent by centrifugation at 1400 rpm for 4 min. Residual concentration of dye in supernatant was estimated spectro photo metrically by monitoring the absorbance at 617 nm (λ_{max}) using a UV-Vis ELICO spectrophotometer (Model SL-177). The

% adsorption rate of MG can be calculated by the equation.

$$\text{\% of dye adsorption} = \frac{C_0 - C_t}{C_0} \times 100$$

-------------------------------- (1)

Where C_o is the initial concentration of dye and C_t is the concentration of dye at any time.

Characterization of PHFAC Adsorbent

The surface morphology of the prepared PHFAC adsorbent was characterized by Scanning Electron Microscopy (SEM, Model: SU-70, Hitachi, Japan). The FT-IR studies before and after the adsorption has been performed on a Perkin Elmer Spectrometer (Spectrum 1000) with KBr pellet technique in the range of 400-4000 cm-1.

Results and Discussions

Morphological Analysis

Scanning electron micrographs of the PHFAC is shown in (Figure 1). It revealed that the morphology of the PHFAC was a no spherical shape i.e. flakes like and has uniform size and distribution.
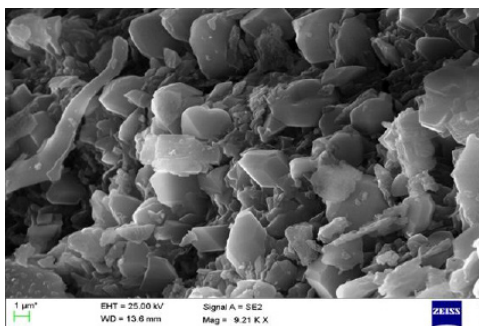

Figure1: SEM Picture of PHFAC.

FT-IR Studies

FT-IR spectra analysis at wave numbers ranging from 400 to 4000 cm−1 of the PHFAC before and after adsorption process were recorded as shown in the (Figure 2) (a,b). (Figure 2a) reveals that there are many functional groups on the surface of the adsorbent. There is a wide adsorption band on fresh adsorbent for example at wave numbers between 3000 and 3755 cm−1 (with a maximum at 3422 cm−1), which is representative of free and bonded hydroxyl groups (-OH) bonded to the surface of fresh adsorbents [17-19]. Moreover, a broad adsorption band at wave numbers ranging 2855 to 3000 cm−1 was observed in the spectrum of fresh adsorbents, demonstrating that C-H groups are present on the surface of PHFAC. Also, a wide adsorption band at wave numbers ranging 1450-1710 cm−1 may be related to the presence of phenolic groups. There is a number peak at wave numbers ranging 1550-1700 cm−1, indicating that the presence of carbonyl groups (C-O) on the surface of fresh adsorbents [20,21].

In (Figure 2b) by comparison of PHFAC after the role of

functional groups is significantly visible. Comparing these two shows that fresh PHFAC peaks after adsorption has decreased significantly that indicating the activity and role of functional groups in MG adsorption exists in solution.
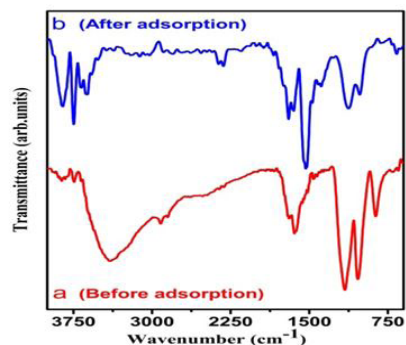

Figure 2 (ab): FT-IR spectra of (Figure 2a) before and (Figure 2b) after adsorption of PHFAC.

Effect of Contact Time

The effect of contact time for 5, 10, 15, 20, 25, 30 min and 5 mg/L dye solution with activated carbon dose (3 mg/L) at acidic pH of solution (pH 1) is shown in (Figure 3). The uptake of MG onto a PHFAC increases with time until the equilibrium is attained between the particles MG adsorbed on the activated carbon and the remaining in solution. It has been observed that the uptake of MG is high in the beginning, followed by a much slower subsequent adsorption and at last leading to steady state. About 80% of the MG was absorbed by PHFAC during first 25 min of the reaction, while only a small part of the additional removal occurred during the rest. This rapid adsorption may be attributed to the external surface adsorption. This suggests most of the adsorption sites of the PHFAC existed in the exterior of the adsorbent and were easily accessible by the MG species resulting in a rapid approach to steady state [22].
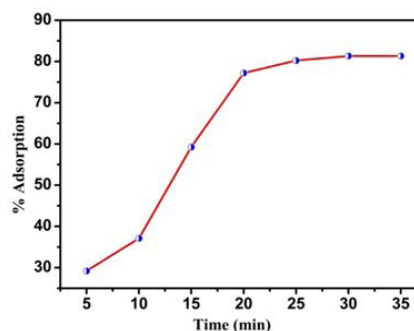

Figure 3: Effect of Contact Time on Adsorption of MG.

Effect of Adsorbent Dose

(Figure 4) shows the effect of adsorbent dose on the % adsorption at equilibrium conditions. It was observed that the amount of MG adsorbed is varied with the varying in the adsorbent dosage.

The amount of MG adsorbed increases with an increase in adsorbent dosage from 1 mg to 4 mg. The percentage of MG adsorption was increased from 80 to 86 %. The increase in the adsorption of the amount of adsorbent is obvious due to increase in surface area. Increase in adsorbent dosage, more than 4 mg the % adsorption decreases, because of saturation of adsorption sites and dye molecules in solution [23].
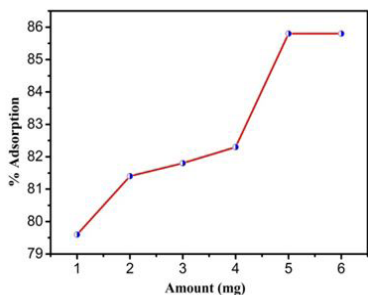

Figure 4: Effect of Adsorbent Dosage on Adsorption of MG.

Effect of pH

The pH of the MG solution plays an important role in the whole adsorption process and particularly on the adsorption capacity, influencing not only the surface charge of the adsorbent, the degree of ionization of the material present in the solution and the dissociation of functional groups on the active sites of the adsorbent [24]. The effect of solution pH (Figure 5) on the amount of MG adsorbed was studied by varying the pH under constant process parameters at equilibrium conditions. From (Figure 5) it was observed that the adsorption was strongly dependent on pH. Experiments were performed by varying pH from 2 to 12 at room and the agitation time was 60 min. The maximum removal efficiency (95%) of PHFAC was found at pH 6 and at higher pH values the removal efficiency decreases gradually.
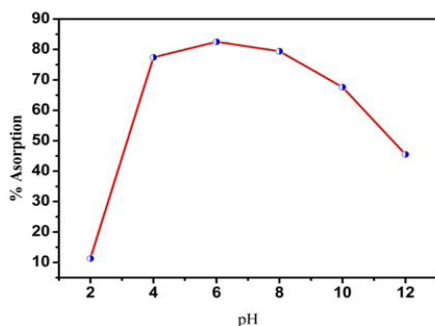

Figure 5: Effect of pH on Adsorption of MG.

Effect of Initial Concentration of Dye

The initial concentration of dye is also another parameter which needs to be taken into account. The adsorption of MG was carried out at different initial concentrations ranging from 5 mg/L to 25 mg/L with 20 min contact time using PHFAC. It is very interesting to note that the percentage of adsorption for 5 mg/L dye

solution was very low since the availability of dye molecules to the adsorbent was poor. With increasing the concentration of dye to 10 ppm, percentage of adsorption slightly increases further for 15ppm of MG concentration, and percentage of adsorption onto PHFAC was found to be high (96%). This experimental result clearly explains that availability of dye molecules to interact with the adsorbent should be in the optimum range. As the initial dye concentration increased from 5 - 10 mg/L, 15 - 20 mg/L, the adsorption of MG onto the PHFAC is indicating that the initial concentration provided a powerful driving force to overcome the mass transfer resistance between the aqueous and solid phases. (Figure 6) shows the effect of initial concentration of the dye.
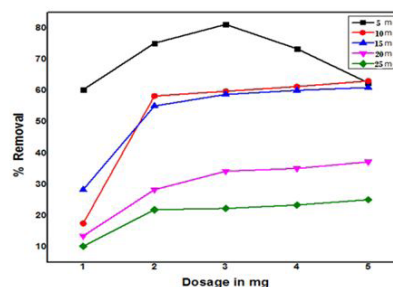

Figure 6: Effect of Initial Concentration of Dye on Adsorption.

Desorption Studies (Regeneration)

0.003 g of PHFAC was added to 50ml of aqueous MG dye solution, after the adsorption studies, the adsorbent was collected by filtration and washed with deionizer 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 and stirred for 6 hrs after which it was centrifuged at 1200 rpm for 6 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 % desorption vs. time in min as shown in (Figure 7). It shows percentage of desorption decreases for every recycling process in the order of Cycle 1 > Cycle 2 > Cycle 3.
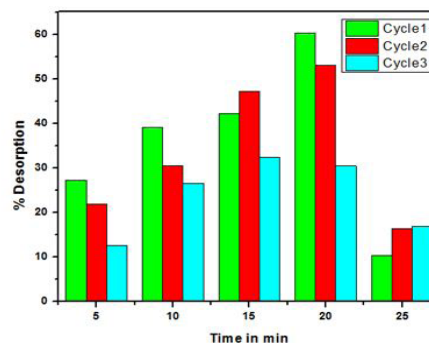

Figure 7: Desorption Plot.

Isotherms Studies
Langmuir Isotherm Model

The Langmuir model is applicable for monolayer adsorption

onto a surface with a finite number of identical sites, [25] where the sorption of each sorbet molecule on to the surface has equal sorption activation energy. The linear form of Langmuir isotherm equation is given as:

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

--- (2)

Where q_e is the amount of chromium adsorbed per unit weight of adsorbent (mg/g), C_e the equilibrium concentration of chromium in the bulk solution (mg/L), qm axis the monolayer adsorption capacity (mg/g) and KL is the Langmuir equilibrium constant (l/mg) related to the adsorption. Both are determined from a plot of C_e/q_e versus C_e (Figure 8).
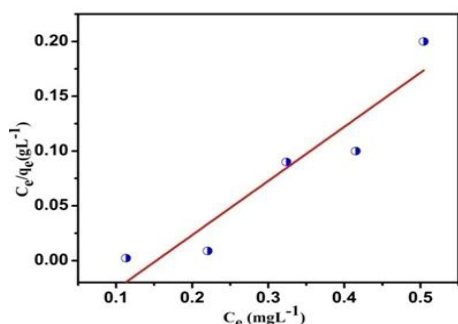

Figure 8: Langmuir Adsorption Isotherm.

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, RL [26], which is defined by the following equation:

$$R_L = \frac{1}{1 + (q_{max} \times k_L) \times C_o}$$

---------------------------------- (3)

Where C_O 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 ($R_L = 0$) [27]. 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-homogenous adsorption.

Langmuir isotherm				Freundlich isotherm		
q_m (mg/g)	R_L (L/mg)	R_2	R_L	K_F (mg/g)	1/n	R_2
2.024	-0.03	0.8629		20.859	0.262	0.9352

Table 1: Isotherm Parameters for Adsorption of MG onto PHFAC.

Freundlich Isotherm Model

The Freundlich isotherm is purely empirical equation and it best describes adsorption onto a heterogeneous surface [28]. The linear form of the Freundlich equation is given as:

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

-------------------------------------- (4)

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 R2 value of the Freundlich plot (Figure 9) is higher than the Langmuir, suggesting that the equilibrium adsorption of MG onto PHFAC 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 MG was favorably adsorbed by PHFAC.
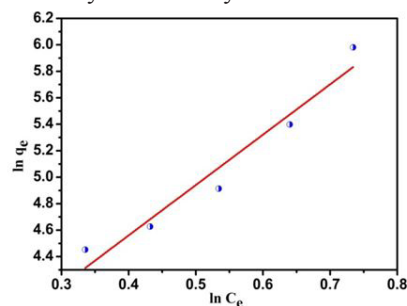

Figure 9: Freundlich Adsorption Isotherm.

Kinetic Studies

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 (Equation 5) was applied to understand the adsorption kinetics quantitatively. This model has been used to calculate the rate constant of adsorption experiments [29].

$$r = -dc/dt = k_r KC/(1 + KC)$$

-------------------------------------- (5)

Where 'r' is the adsorption rate, 'kr' is the adsorption rate constant, 'K' is the absorption coefficient of the reactant, and 'C' is the reactant concentration. When C is very small, (Equation 5) can be expressed by (Equation 6).

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

-------------------------------------- (6)

Where 'k' is the first-order rate constant. Set t=0, C = C0, (Equation 7) can be induced.

$$ln\, Co/C = kt$$

-------------------------------------- (7)

(Figure 10) shows the adsorption rate of PHFAC under room temperature. It is clear that the kinetic simulation curve with stirring time (t) as abscissa and ln (C_0/C) as the vertical ordinate is close to a linear curve with the fitting constant 'R' greater than 0.96. It is observed that PHFAC exhibit good adsorption activity (k = 0.04 min-1).
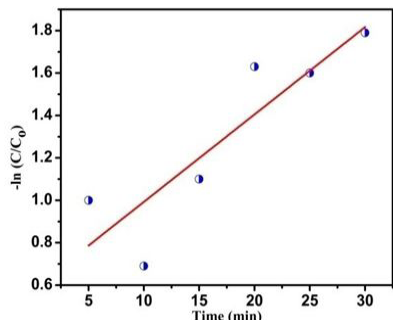
Figure10: First Order Kinetic Plot.

Pseudo Second Order

Pseudo-second order kinetics [30] is expressed as follows,

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

-------------------------------------- (8)

Where k_2 is the rate constant (g/mg min). Integration of (Equation 8) at the boundary, qt= 0 at t = 0 and qt= qt at t = t and then rearrangement to a linear form gives (Equation 9).

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

-------------------------------------- (9)

The value of k2 and qe can be determined from the slope and the intercept of the plot t/qt vs. t, respectively. As can see from (Table-2) (Figure 11), time-concentration data were best fitted to all used kinetic models with higher correlation coefficient onto pseudo second order kinetic model.

First order kinetics		Pseudo second order kinetics		
K_1 (Sec^{-1})	R^2	q_e (mg g^{-1})	K_2 (Sec^{-1})	R_2
0.04	0.7364	4.37	0.394	0.9325

Table 2: Kinetics Model of Adsorption of MG onto PHFAC.
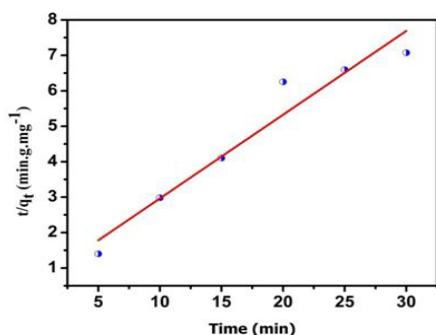

Figure11: Pseudo Second Order Kinetic Plot.

Conclusions

The present study gives the evidence of the possible benefits of using the dry biomass of PHFAC for the removal of MG from aqueous media and the biomass concentration was found to

have a significant impact. The dried activated biomass in the batch experiment was found to be very efficient in removing MG dye from aqueous solution. The maximum removal rate was achieved within 25 min of contact time. The process is strongly affected by several parameters such as: contact time, initial dye concentration and pH of the solution. This study indicated the scope for developing an appropriate technology for effective designing of a waste water treatment plan. However, further work should be performed in order to have a better understanding of MG adsorption binding mechanism by dried PHFAC. It is concluded that this adsorbent has a great potential for removing MG from aqueous solutions as an eco-friendly process.

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