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Utilization of agricultural waste for the removal of congo red, the environmental pollutant from simulated aqueous solutions

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ABSTRACT

Agricultural waste-based adsorbents are becoming attractive choice for the removal of environmental pollutants from water due to their abundance and low cost. Batch adsorption studies were carried out with *Parthenium hysterophorus* L. (an invasive weed) to explore its potential for the removal of the anionic dye (Congo Red, CR) from simulated aqueous solutions. The experiment was conducted to study the effect of different parameters including contact time, pH (3-10), initial concentration and adsorbent dose on dye removal efficiency of the adsorbent. It was found that using the bioadsorbent, the sorption equilibrium for the dye could be achieved within 45 minutes at lower concentrations. The experimental data was analyzed through Freundlich and Langmuir adsorption isotherms. The data fitted better in Langmuir isotherm (R^2=0.975). The results showed that the maximum adsorption capacity (Q_m) is 14.49 mgg^{-1}. The results so obtained indicate that the bioadsorbent can be used efficiently for the dye removal.

Key words: Adsorption, Bioadsorbent, Contact time, Equilibrium, Langmuir isotherm

Amongst different chemicals utilized in different industries of cosmetics, rubber, paper, food and textile industries, dyes and pigments are commonly used for giving desirable color to the product (Alzaydien 2009). According to the available data more than 10000 dyes are commercially available worldwide with the production of over 7×10^5 metric tonnes and consumption of more than 10^7 kg per annum (Robinson *et al.* 2001). As these dyes have high solubility in water and easy procedures to dye fabrics, these are used by the textile industry in majority (about 90%). During the dyeing process, 10-15% of the dye is lost which is discharged with the effluents resulting in contamination of water resources (Mohammed *et al.* 2014). The addition of even small amount of dyes can affect physico-chemical properties of water bodies. This leads not only to undesirable colored water but also disturbs biological activity of aquatic organisms through interference in the penetration of sunlight thus disturbing their population and aquatic dynamics. Hence it is desirable to remove dyes from water sources.

Congo Red (Disodium-4-amino-3-[[4-[4-[(1-amino-4-sulfonatonaphthalen-2-yl) diazenyl]phenyl]phenyl]diazenyl] naphthalene-1-sulfonate) is benzidine based anionic dye that gets metabolized to benzidine which is widely reported to be a human carcinogen (Brown 1987, Rao and Rao 2016). It has also been found responsible for reduction in serum protein concentration which can lead to platelet aggregation, thrombocytopenia and disseminated micro embolism (Frid *et al.* 2007). Synthetic dyes including Congo Red, have complex aromatic structures which make them difficult to biodegrade (Dawood and Sen 2012). Due to their high stability and low biodegradability, conventional biological treatment methods for removal of dyes are not sufficient (Mondal 2008). Therefore different methods have been used for the treatment of the textile effluents as alternative to the above method. These methods include coagulation/flocculation, oxidation, photo catalytic degradation and adsorption (Stammati *et al.* 2005). Amongst these methods, adsorption has been found to be an effective method in terms of simplicity, high treatment efficiency, cost-effectiveness and quick lowering of dissolved dyes concentration (Srivastava *et al.* 2004).Commercially activated carbon has been found to be the most effective adsorbent but the high cost of the product and poor regeneration has paved the way for the researchers to explore the possibilities of alternative adsorbents which are equally effective but cheap.

Parthenium (*Parthenium hysterophorus* L.), also known as Congress grass, ramphool, star weed or white top, is a herbaceous plant which reaches to a height of 2m and within 4-6 weeks of germination it starts flowering (Rao 1995). It is known to cause several health problems to humans. It is ever growing unwanted weed which is available in abundant quantity round of the year. It is also reported to decrease agricultural productivity so it becomes utmost important

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to manage the weed present in the nature. In Ethiopia, *P. hysterophorus* is reported to cause sorghum grain yield losses between 40-97% if it is left uncontrolled throughout the season (Tamado *et al.* 2002).

The aim of this study was to explore the adsorption potential of the weed for the removal of Congo Red dye from aqueous solution under different experimental conditions. Langmuir and Freundlich models were used to fit the experimental data.This will not only help to explore the possibility of low cost alternative to activated carbon but also the utilization of the notorious weed in a better way.

MATERIALS AND METHODS

Preparation of adsorbent

To conduct the experiment, plants of *Parthenium hysterophorus* weed (PH) were collected locally from IARI field side and roadside randomly. The samples were mixed to get uniform composition of the adsorbent. The samples were washed with water to remove any adhering dirt, cut into small pieces and dried in sunlight to get the moisture evaporated. The material was then oven dried at 105°C for 24 h to ensure complete removal of water and get constant weight. The dried material was then ground in steel blender and sieved through BSS 30 sieve to get uniform particle size of 500 microns. The material was stored in glass bottle and kept in desiccator. The adsorption experiment was carried out with the same material without doing any chemical or physical treatment.

Adsorbate

Chemicals used for the study including Congo Red dye H_2SO_4, NaOH were supplied by Merck Co. (M) Germany. The physico-chemical properties of the dye are given in Table 1. To prepare the stock solution of 1000 mgL^{-1}, known amount of the dye was weighed and dissolved in double distilled water. Experimental solutions of desired concentrations (10, 25, 50, 60, 80 and 100mgL^{-1}) were obtained by successive dilutions of the stock solution.

Characterization and analysis

Fourier transform infrared (FTIR) spectroscopy (Model-Bruker Alpha, Germany) was used to detect the surface functional groups of PH. The spectrum was recorded from 4000 – 600 cm^{-1}.Elemental analysis of the adsorbent was done on CHNS analyzer (Eurovector, Model E 83000, Italy). The surface morphology of the adsorbent before and after adsorption was studied using Scanning Electron Microscope (Model-Tescan Vega3, Czech Republic). The concentration of the CR dye in the solutions before and after adsorption was recorded through UV-Visible spectrophotometer (MS-UV 11, India) at a maximum absorbance wavelength of 497 nm (λ_{max}).

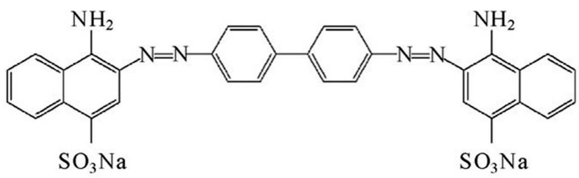
Effect of contact time

To assess the effect of contact time on dye adsorption was performed by taking 1.5gL^{-1} in each of the 250 mL conical flasks. 50 mL of the dye solution of 10 mgL^{-1} concentration was added in the flasks. All the flasks tightly covered and agitated on mechanical shaker keeping other parameters (pH 7 and adsorbent dose 1.5 gL^{-1}) constant. The samples were withdrawn at pre determined time intervals (15, 30, 45, 60, 75, 90, 105 min.). The concentration of the supernatant was measured at 497 nm using UV- visible spectrophotometer.

Effect of adsorbent dose

The effect of adsorbent dose on dye removal was

Table 1 Physico-chemical properties of Congo Red Dye

Dye name	Congo Red (Syn. Cosmos Red, Cotton Red B, Cotton Red C, Direct Red 28, Direct Red R)
IUPAC name	Disodium-4-amino-3-[[4-[4-[(1-amino-4-sulfonatonaphthalen-2-yl) diazenyl]phenyl]phenyl] diazenyl]naphthalene-1-sulfonate
Chemical formula	$C_{32}H_{22}N_6Na_2O_6S_2$
Molecular weight	696.664 $gmol^{-1}$
Colour Index Number (CI No.)	22120
Color	Brownish-red powder; yellowish-red in water, orange in ethanol
λ_{max}	497 nm
Melting point	>360°C
Hazardous Decomposition Products	Nitrogen oxides (NO_x), Carbon monoxide (CO), Carbon dioxide (CO_2), Sulfur oxides,Ammonia
Hydrogen Bond Donor Count	2
Hydrogen Bond Acceptor Count	12
Solubility in solvents	1.16×10^5 mgL^{-1}at 25°C in water, 1.0 $mgmL^{-1}$ in ethanol
Structure	

studied by using different amount of adsorbent material (1.5, 2.5, 3.5, 5, 7.5 and 10 gL^{-1}) without changing the pH of the solution. All the flasks containing different doses of adsorbent and 50 mL of 10 mgL^{-1} dye solution were agitated on mechanical shaker at constant speed of 130 rpm at 25°C.

Effect of pH

To study the effect of pH on CR removal, the experiment was carried out at different pH ranging from 3-10. For the purpose, flasks containing 50 mL of dye solution of 10 mgL^{-1} concentration at different pH (3,4,6,7,8 and 10) were agitated with the adsorbent dose of 5 gL^{-1} in shaker at 25°C. The pH of the solution was adjusted with 0.1N NaOH and 0.1N HCl solutions using the pH meter.

Batch mode adsorption experiment

Batch adsorption studies were carried out to study the effect of individual parameters such as adsorbate concentration, adsorbent dose, contact time and pH. The studies were carried out by agitating fixed amount of sorbent (5gL^{-1}) with 50 ml of dye solutions of desired concentrations (10-100 mgL^{-1}) without changing pH (4±0.2) at 25°C temperature in the conical flasks with glass stopper. The flasks were agitated at 130 rpm for 120 min to ensure equilibrium on mechanical shaker. Using a double beam UV-Vis spectrophotometer, the dye concentration was estimated in supernatant at time t=0 and equilibrium at 497 nm wavelength. The amount of dye adsorbed at equilibrium, q_s (mgg^{-1}) was calculated with the following equation

$$q_s = \frac{(C_o - C_e)V}{W} \quad (1)$$

C_o and C_e (mgL^{-1}) are initial and equilibrium concentration of dye respectively, V is the volume of solution (L) and W is the mass of adsorbent (g). The removal efficiency (%) was calculated

$$\text{Removal efficiency} = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

where C_o and C_e are initial and equilibrium concentration of dye being studied, respectively. Each experiment was conducted in triplicates along with the blank runs of only adsorbent in 50 mL of double distilled water and 50 mL of dye solution without any adsorbent.

Theory

The experimental data of C_e values for different initial concentrations were fitted into the Langmuir and Freundlich isotherms models for the different initial dye concentrations to test the adsorption mechanism.Langmuir isotherm assumes monolayer coverage of the adsorbent surface by the adsorbate molecules. The linear form of Langmuir equation (Langmuir 1918) can be represented as following

$$\frac{C_e}{q_e} = \frac{1}{(bQ_m)} + \frac{C_e}{Q_m} \quad (3)$$

where, C_e (mgL^{-1}) is the equilibrium concentration, q_e (mgg^{-1}) the amount of adsorbate adsorbed per unit mass of adsorbate, and Q_m and b are the Langmuir constants related to adsorption capacity and rate of adsorption, respectively.

On the other hand, Freundlich isotherm assumes that heterogeneous layers of adsorbate can be formed and to explain the phenomenon, an empirical model can be used (Freundlich 1906). The equation can be expressed as:

$$\ln q_e = 1/n \ln C_e + \ln K_F \quad (4)$$

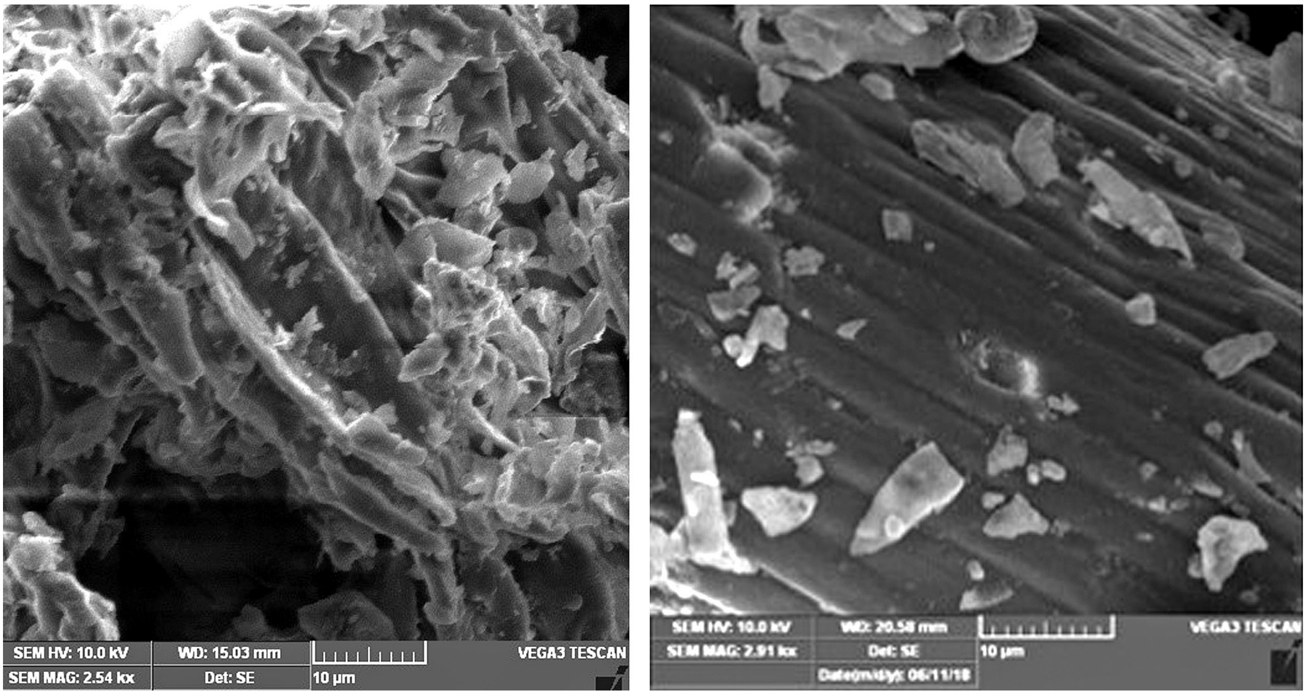


Fig 1 SEM images of PH adsorbent before and after dye adsorption.

where, K_F is the adsorption capacity and 1/n implies sorption intensity.

Data analysis

The data generated was analyzed by using Microsoft Excel to compute mean, standard deviation and linear regression values.

RESULTS AND DISCUSSION

Characterization

To study the presence of various functional groups present on the surface of the biosorbent and their role in adsorption, FTIR spectrum of the biosorbent was obtained and analyzed. The peak observed at 3447 cm^{-1} corresponded to the presence of amino and hydroxyl groups on the adsorbent (Kousha *et al.* 2015). The presence of aliphatic fraction was represented by peak at 2870 cm^{-1} while peak at 1735 cm^{-1} and 1422 cm^{-1} indicated the presence of C-X and C-F group respectively. The peak at 1322 cm^{-1} showed C-N stretching vibration whereas peak at 1022 cm^{-1} could be associated with C-O-C functional groups (Coates 2000).

The elemental analysis of the PH biomass showed that it contained C(%): 35.47, N(%): 2.16, S(%): 0.162, H(%): 6.26, O (%): 55.94, O/C: 1.578, H/C: 0.176.

Fig 1 shows SEM image of the adsorbent before and after dye adsorption. It could be seen from the Fig 1(a) that the surface of *Parthenium hysterophorus* was heterogeneous. The porous structure of the adsorbent could be clearly seen which indicated that there is a good possibility to trap and adsorb the dye molecules. Fig 1b showed that after dye adsoption the surface of the adsorbent was smooth which gave indication that it was covered by the dye molecule after the adsorption on the surface.

Effect of contact time

The effect of contact time on the removal of CR dye by unmodified *Parthenium hysterophorus* (Congress grass) at different equilibrium time is given in Fig 2. The results indicate that the dye uptake increased with time initially and reached equilibrium state. At the initial stage of adsorption, the process was rapid which slowed down subsequently while approaching the equilibrium. The adsorption rate was higher initially because of availability of large surface area for adsorption of anionic dye (Hameed and Ahmad 2009).

With the passage of time, number of active sites decrease so there is slight increase in dye uptake. For the contact time studied, it was found that 45 min. of agitation time is sufficient for achieving equilibrium for 10 mgL^{-1} dye concentration. The equilibrium time for the same dye can vary with different adsorbents. Foroughi-Dahr *et al.* (2015) showed that equilibrium time for CR adsorption on tea waste was 150 min. up to the concentration of 150 mgL^{-1} while equilibrium for the same dye reached within 90 min. with adsorption efficiency of 96% when roots of *E. crassipes* were used in another study (Wanyonyi *et al.* 2014).

Effect of adsorbent dose

The dose of an adsorbent affects its adsorption capacity so plays a pivotal role in adsorption process. In order to evaluate the effect of adsorbent dosage on the removal of CR dye the dose between 1.5 gL^{-1} to 10 gL^{-1} was varied. The percentage removal of dye at different doses is given in Fig 3. It can be seen that as the adsorbent dose increased until 5gL^{-1}, the removal of CR dye increased. The increase was from 73.45 to 95% as the adsorbent dose increased from 1.5 to 5 gL^{-1}. This can be attributed to the increase in available surface area of the adsorbent which results in availability of more adsorption sites. As the adsorbent dose increased from 5 to 10 gL^{-1}, percentage removal of the dye remained almost constant with a slight decrease which can be attributed to the fact that if too much adsorbent is added it can hamper the transportation of dye ions to the active adsorption sites. It also implies that even after increasing the amount of adsorbent the complete dye removal cannot be achieved so the optimum dose for carrying out the adsorption of the dye is 5 gL^{-1}(Abdur Rahman *et al.* 2013).

Effect of pH

pH plays a crucial role in controlling the adsorption behavior as it affects both aqueous chemistry and surface

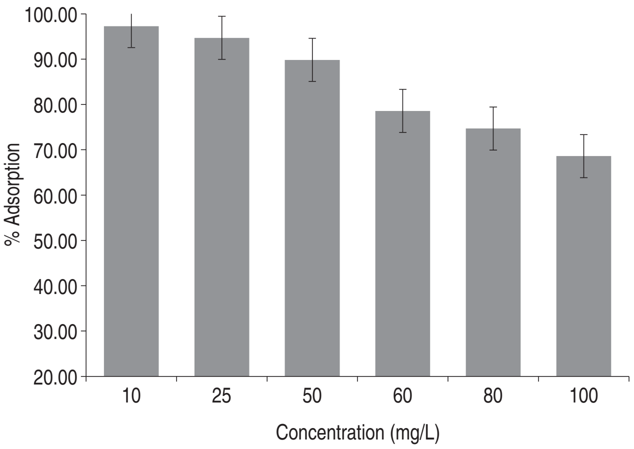


Fig 2 Per cent removal of CR dye through adsorption by *Parthenium hysterophorus* (5 gL^{-1}; contact time: 100min: pH 4 at different concentrations).

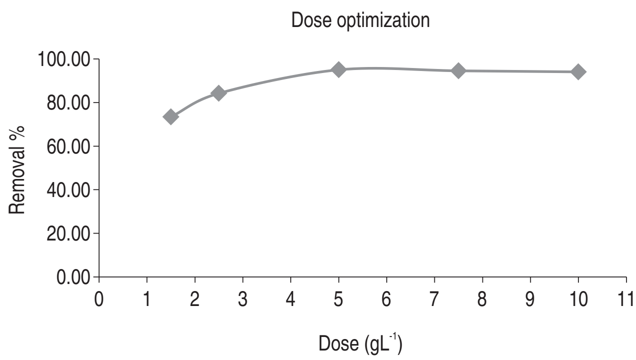


Fig 3 Effect of adsorbent dosage on CR adsorption by *Parthenium hysterophorus* (pH 4: contact time: 100min, 10 mgL^{-1}).

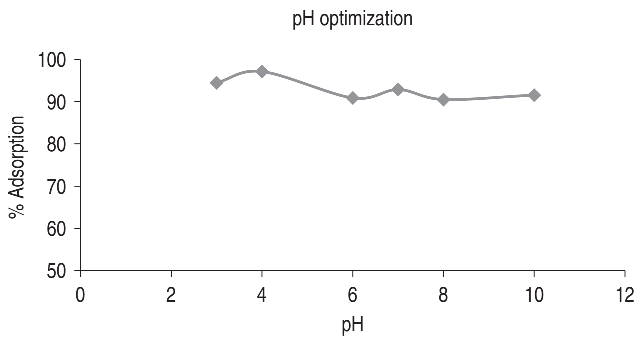


Fig 4 Effect of initial pH on CR adsorption by *Parthenium hysterophorus* (adsorbent dose: 5 gL-1; dye concentration 10 mgL-1; Contact time:100 min).

binding sites of the adsorbent. The effect of pH on the removal of CR dye is shown in Fig 4. The experiment was carried out at 10 mgL^{-1} dye concentration and 5 gL^{-1} adsorbent dose and effect of pH was studied within pH range of 3-10. Though there are several studies which studied the effect of pH on the adsorption of the dye but the results seem to contradict each other (Liu *et al.* 2012, Khaniabadi *et al.* 2017).

In the present study, the per cent removal of dye solution increased from 94 to 97 when the pH was increased from 3 to 4. After that up to pH 10, the per cent removal remained almost same ranging from 90-92%. This can be attributed to the fact that at low pH high adsorption results because of high electrostatic interaction between anionic dye and positively charged surface of adsorbent. So the optimum pH for carrying out the adsorption experiment was taken as 4.

Adsorption experiment

The adsorption of CR was studied by varying the dye concentration from 10-100 mgL^{-1}. The percentage adsorption was found to be 97% being the highest at 10 mgL^{-1}. As the initial dye concentration increases, there is an increase in the adsorption of the dye per unit mass of the adsorbent (Hameed and Ahmad 2009, Foroughi-Dahr *et al.* 2015). In the study it was found that the equilibrium amount of CR dye so removed showed an increase from 1.94 mgg^{-1} to 13.72 mgg^{-1} with the increase in the initial dye concentration from 10 to 100 mgL^{-1}. It can be attributed to the driving force of the initial dye concentration which helps to overcome the resistance for the mass transfer between two phases involved in the process (Srivastava *et al* 2006).

Adsorption isotherms

An adsorption isotherm is used to describe the retention/ mobility of the molecules between two phases (liquid-solid) when the equilibrium state for adsorption is achieved (El-Geundi 1991). Therefore to determine the application of adsorption to remove CR dye from aqueous solutions by PH biomass and to find suitable model for the purpose, the isotherm data was fitted to different isotherm models. In the present study, Freundlich and Langmuir isotherms models were used. In Freundlich model, the graph was plotted between ln q_e and ln C_e. The linear graph (Fig 6) confirmed the applicability of the model.The isotherm plotted between ln q_e and ln $C_e$was used to calculate the heterogeneity factor (n) that indicates whether the nature of adsorption is linear (n = 1), chemisorption (n < 1), or a physisorption (n > 1). This parameter can also help to identify if the process followed normal Langmuir isotherm (1/n < 1).On the basis of the data obtained, the values of n and 1/n were found to be 2.53 and 0.395, respectively.

The Langmuir isotherm assumes that adsorption takes place on a homogeneous surface and all points are equally available for interaction with the adsorbate. A straight line was obtained on plotting graph for C_e/q_e against C_e with $1/Q_m$ as slope (Fig 5). The Langmuir constants *b* and Q_m were calculated from the slope and the intercept of the plot and their values are listed in Table 2. The correlation coefficient of the isotherm (R^2) was found to be 0.975 indicating the validity of the isotherm.

It could be seen from Fig 5 and Fig 6 that the experimental data fitted better in normal Langmuir isotherm which indicated that the nature of adsorption is physisorption and there is monolayer coverage of the dye molecules on the surface of PH biomass.Table 2 shows the comparison of maximum adsorption capacity of CR dye on different adsorbents. The data indicates that the PH biomass used in the study has good adsorption capability of 14.49 mgg^{-1} if

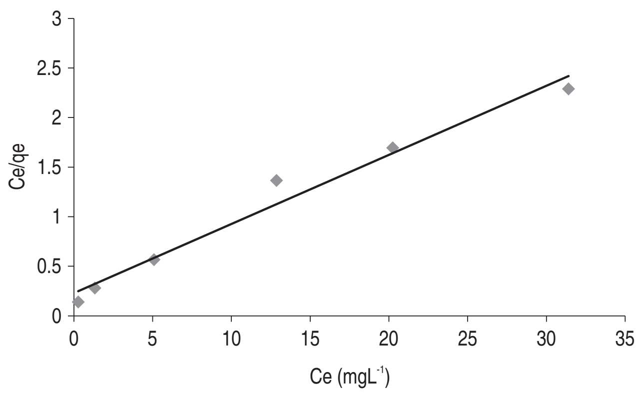


Fig 5 Langmuir isotherm for CR dye adsorption on *Parthenium hysterophorus*.

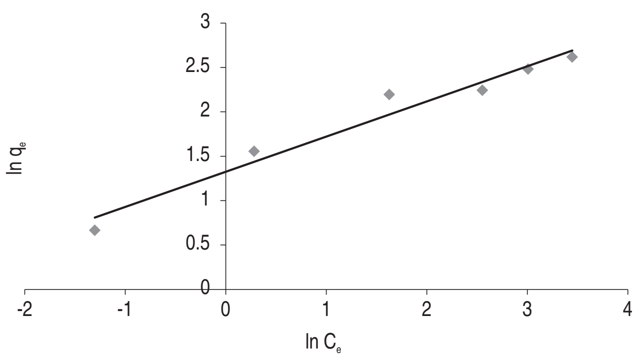


Fig 6 Freundlich isotherm for CR dye adsorption on *Parthenium hysterophorus*.

Table 2 Isotherm parameters for removal of CR dye on PH biomass

Langmuir			Freundlich		
Qm (mgg^{-1})	b	R^2	K	n	R^2
14.49	0.302	0.975	3.76	2.53	0.962

Table 3 Comparison of the maximum monolayer adsorption of CR onto various adsorbents

Adsorbents	q_m (mgg^{-1})	References
Cashew nut shell	5.18	Kumar *et al.* 2010
Pomegranate	10	Ghaedi *et al.* 2012
E. crassipes roots	1.580	Wanyonyi *et al.* 2014
Tea waste	23.26	Foroughi-Dahr *et al.* 2015
Parthenium hysterophorus	14.49	Present work

comparison between different adsorbents is done which is available in literature.

Conclusion

The study was carried out with whole plant biomass of *Parthenium hysterophorus* as adsorbent for the removal of Congo Red dye from the aqueous solutions. The results showed that the amount of dye adsorbed on the adsorbent depends on pH, contact time and adsorbent dosage along with the initial dye concentration. The equilibrium for the adsorption can practically be achieved in 90 min. The experimental data fitted well in both Freundlich and Langmuir adsorption isotherm models. However Langmuir isotherm showed a bit better fit than Freundlich model with R^2 value of 0.975 indicating the homogeneous nature of the adsorbent. The maximum adsorption capacity (Q_m) was found to be 14.49 mgg^{-1} at pH 4.These results show that *Parthenium hysterophorus* has the potential to be used as low cost adsorbent for waste water treatment that can also pave the way for effective management of weed population.

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