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Phytoremediation Potential of *Hibiscus rosa-sinesis* for Removal of Methylene Blue Dye and its Kinetic, Adsorption Studies in Aquatic System

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Phytoremediation technology emerges as a green, environmentally friendly and cutting-edge technology in last ten decades, which provides an economical solution for removing dyes from wastewater. In current study, the terrestrial plant *Hibiscus rosa-sinesis* L. explored for remediating the triarylmethane dye as methylene blue including the various parameters *viz*. initial dye concentration (10 mg L^{-1}), pH (3-10), contact time (8 to 40 h) from synthetic wastewater. On assessment of these various parameters, the maximum percentage decolourizations were recorded as 86 (10 mg L^{-1}) and 75 (20 mg L^{-1}) at favourable pH 6. The kinetics studies were best fitted to pseudo-first order with the correlation value $R_2 \geq 0.96$. The Langmuir adsorption isotherm proved more favourable with $R_2 \geq 0.99$. Fourier transform infrared spectroscopy (FTIR) confirmed the adsorption of methylene blue dye by *H. rosa-sinesis*. Hence, this plant can be used for remediation of dyes from wastewater.

Keywords: Phytoremediation, Dye decolourization, Terrestrial plant, Langmuir Adsorption isotherm, Kinetic studies.

INTRODUCTION

Xenobiotic organic compounds are the foremost components of water contamination for enhancing environmental pollution. The major source of xenobiotic pollutants are the dyes and their presence in the aquatic life is the vast apprehension due to their linkage with toxic, mutagenic, carcinogenic and genotoxic properties. These dyes are also showing the harmful effects on the natural stability of the water ecosystem, which leads to reduce the oxygen level of the water bodies [1].

There are approximately 8×10^5 tons annually synthetic dyes produced which are used in fabric industries. However, 10-15% of dyes are thrown by textiles industries without treatment in their surroundings areas [2]. This causes harsh nuisance to the aquatic life, which results in damaging the food web and aesthetic appeal of the environment. The dyes interfere with microbial development and impede aquatic plants' ability to photosynthesize by absorbing and reflecting sunlight in water. Dye exposure can result in allergic dermatitis, skin rashes, cancer, genetic mutations, *etc*. Due to the dyes intricate aromatic structures, which render them useless in the presence of heat, light, microorganisms and oxidising agents and make dye breakdown a challenging issue [3]. These problems pose a major risk to both human health and water quality, making them an urgent topic of concern. Therefore, science and technology should take initiative to purify wastewater by creating efficient dye removal methods. For the treatment of wastewater containing dyes, a variety of physical and chemical techniques are utilized, such as filtration, sedimentation, chlorination, adsorption, flocculation, coagulation, electro-coagulation, osmosis, *etc*. But these techniques are costly, not safe, less effective, secondary pollutant producers and form a high amount of sludge. The different effective biological methods have been used to overcome the problem faced by the physico-chemical methods for the removal of textile dyes from the wastewater [4]. The phytoremediation is an innovative, ecologically sustainable, inexpensive, aesthetically acceptable and green approach by which plants are often removing the contaminants from soil, water and air [5]. Various types of plants were examined for the phytoremediation of dyes, from which the aquatic plants such as *Salvinia molesta* Mitchella [6], *Pistia stratiotes* L. [7], *Lemna minor* L. [8], *Chara vulgaris* L. [9] were also studied. Many terrestrial plants species *viz*. *Phragmites australis* (Cav.) Trin.ex.Steud [10], *Typha angustifolia* L. [11], *Portulaca grandiflora* Hook [12] and *Zinnia angustifolia* Kunth [13] were analyzed as potential to degrade the acid orange 7, reactive blue 19, navy blue HE2R, remazol black B dyes, respectively. *Aster amellus* L. was utilized to remove the dye remazol red [14], *Ipomoea hederifolia* L. [15], A*lcea rosea* [16], *Bacopa monnieri* [17] are some other recently identified terrestrial species for remediation of harmful dyes. *Hibiscus rosa-sinesis* L. is another common terrestrial plant, which is still unexplored for its potential for phytoremediation of dyes in water system. In literature, only work reported on *H. rosa-sinesis* as bioadsorbent in its non-living form for removal of reactive dyes [18,19]. Therefore, it is decided to explore the potential of *H. rosa-sinesis* as living plant to remove the toxic methylene blue dye from aquatic system in this research work. The various kinetics and adsorption isotherms experiments were also conducted to recognize the dye remediation mechanism by plant in living conditions.

EXPERIMENTAL

Methylene blue dye with chemical formula $C_{16}H_{18}N_3SCl$ and molecular weight 319.85 g mol^{-1}, with the highest purity analytical grade of Merck Ltd., India was utilized for the experimentation. The dye is water-soluble therefore stock solution (250 mL) of methylene blue dye had been prepared by addition of 250 mg dye in 250 mL of distilled water and thereafter, the required concentrations of dye solution were prepared with dilution of stock solution with distilled water.

Plant material: The plants of *H. rosa-sinesis* of equal size were collected from Guru Teg Bahadur College, Amritsar (India). The plants were selected for present study with the length of 107 cm, width of 4-5 cm, weight 30 g and 1 month old plant. After plant collection, they were cleaned properly with tap water and subsequent with distilled water to ready the plant for experimentation. When the soil particles were completely removed from the plant, then plant growth was tested in Hoagland nutrient solution for 10 days so that the plants could be able to accommodate for medium changes from the soil to aqueous [20]. The growth of plants was observed after 1 week. Later, the plants were placed into 250 mL beakers having 100 mL dye solution with supportive system for plants.

Experiment design: The preliminary experiments were performed with 100 mL of 10 and 20 mg L^{-1} methylene blue dye solution in 250 mL beakers by treating with 30 g (wet wt.) of fresh plant. Further, the experiments were conducted with different concentrations of methylene blue dye as 0 (biotic control), 10, 20, 30, 40 and 50 mg L^{-1} by using 100 mL volume of dye solutions with 30 g *H. rosa-sinesis* (approx. wet wt. of each plant) to analyze the impact of initial dye concentration and contact time on decolourization potential and amount of dye adsorbed by the plant. In similar pattern, abiotic controls were also maintained for each tested concentration. The impact of pH on the decolourization pattern of methylene blue dyes onto *H. rosa-sinesis* surface were determined with 100 mL of 20 mg L^{-1} of methylene blue dye solution. The pH was determined with the help of 211 Hanna instrument pH meter. The continuity of experiments was carried up to the equilibrium point where no further significant dye removal was observed by the plant.

UV-visible analysis: A 2 mL concentration of the treated dye samples was taken every 8 to 40 h for determination of the absorbance value by using UV-visible double beam spectrophotometer (Shimadzu-2550). The calibration curve was predicted with the maximum absorbance for methylene blue dye at λ_{max} 665 nm. The decolourization percentage value was computed using eqn. 1 [21]:

$$\text{Decolourization (\%)} = \frac{\text{Abs}_{\text{initial}} - \text{Abs}_{\text{final}}}{\text{Abs}_{\text{initial}}} \qquad (1)$$

The q_t and q_e (mg g^{-1}) dye removal quantity by *H. rosa-sinesis* at any time t and at equilibrium were estimated with C_0, C_t and C_e the initial dye concentration, concentration any time t and the concentration at equilibrium, respectively in mg L^{-1} using the eqns. 2 and 3 [22-24].

$$q_t = \frac{(C_o - C_t)}{W} \times V \qquad (2)$$

$$q_e = \frac{(C_o - C_e)}{W} \times V \qquad (3)$$

where 'W' is the wet weight of plant taken in g and 'V' is the volume of solution in mL. The triplicate number experiments were performed and controls were maintained without addition of plants in dye solutions.

FTIR analysis: The FTIR spectral analysis was performed to confirm methylene blue dye adsorption by roots of *H. rosa-sinesis* before and after treatment with methylene blue dye. Both control and treated roots were dried up in oven at 60 ºC for 24 h. Later, the dried-out plant was ground with KBr in the ratio of 5:95 to a well powder form. Afterward, the samples were analyzed by means of FTIR spectrophotometer (Agilent-CARRY–630) with range of 4000-400 cm^{-1} [25].

Kinetic studies: The pseudo-first and pseudo-second-order and Elovich equations has been employed to demonstrate the kinetic reaction mechanism. Eqns. 5 and 6 represent the pseudo-first-order equation and pseudo-second-order equation, respectively [26].

$$\log (q_e - q_t) = \log q_e - K_1 t \qquad (4)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \qquad (5)$$

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln \qquad (6)$$

where 'K_1' and 'K_2' are the rate constants in pseudo-first-and pseudo-second-order reactions respectively. The Elovich equation (eqn. 6) determines the rate constant 'α' which correlate the chemisorption and the 'β' value depict the amount of dye adsorption. The R_2 values were employed to predict the best fit for kinetic equation models and to estimate the rationality of the kinetic model. The R_2 values were calculated by plotting t *vs.* log (q_e – q_t), t *vs.* t/q_t and q_t *vs.* ln t for pseudo-first order, pseudo-second order and Elovich equation, respectively. The reaction rate coefficient (K_1 and K_2) was computed from the slope getting the values from the different models. The value of normalized standard deviation ($\Delta q_t\%$) for kinetic models signifies the deviation for the experimental data with theoretical results. The value of normalized standard deviation (%) for each model was calculated by eqn. 7 [27,28]:

$$\Delta q_t\% = 100\frac{\sqrt{\sum\left|\frac{q_{t(exp)} - q_{t(theor)^2}}{q_{t(exp)}}\right|}}{n-1} \quad (7)$$

where $q_{t(exp)}$ corresponds to the experimental value and $q_{t(theor)}$ belongs to the theoretical value for remediation of the dye, 'n' represents the numeral counting of experimental data.

Adsorption isotherms: The Langmuir and Freundlich adsorption isotherms were used to predict the equilibrium study for removal of methylene blue dye by *H. rosa-sinesis* under the phytoremediation process. The linear operational form of Langmuir and Freundlich isothermal models were interpreted by using eqns. 8 and 9, respectively [29,30].

$$\frac{C_e}{q_e} = \frac{1}{bQ_o} + \frac{1}{Q_o}C_e \quad (8)$$

$$\log q_e = \log k_f + \frac{1}{n}\log C_e \quad (9)$$

The adsorption constants 'b' and 'Q_o' of Langmuir equation are associated to the enhanced capacity of dye adsorption on equilibrium and adsorption rate, respectively. The spontaneity nature of dye molecules founds by using the value of 'n' in the Freundlich adsorption isotherm and 'K_f' is the capacity of adsorption by the plant. The different parameters of the Langmuir adsorption isotherm computed with eqn. 10 [23,31]:

$$R_L = \frac{1}{b + C_o} \quad (10)$$

The type of the process is determined by computing the R_L value. For the irreversible and favourable conditions, the value of R_L value comes to zero and if the process is unfavourable the value obtained is greater than one.

RESULTS AND DISCUSSION

The *H. rosa-sinesis* showed an efficient plant to remove methylene blue dye from its synthetic wastewater solution within 40 h of experiment. It was also examined that plant shows similar progression rate in control and after the treatment of dye solution up to three cycles. It was almost 70% decolourization detected in methylene blue dye solution in 24 h of preliminary experiment and equilibrium stage was obtained at 40 h of the reaction. After that, further reactions were conducted to predict the effects of different parameters, kinetics and absorption mechanism of *H. rosa-sinesis*.

Effect of dye concentration and contact time: The plot of amount of dye adsorbed and decolourization percentage with respect to time at different dye concentrations in case of methylene blue is shown in Figs. 1 and 2, respectively. By increasing the concentration of methylene blue dye, it was observed that the decolourization potential of *H. rosa-sinesis*

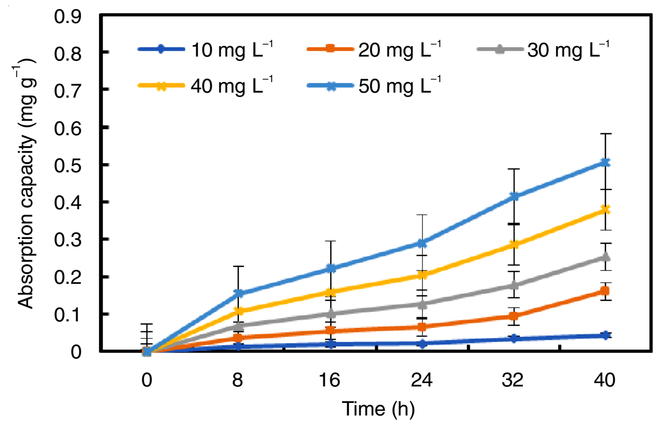

Fig. 1. Plot of adsorption capacity *vs.* time at different concentration on removal of Methylene blue dye by *H. rosa-sinesis*

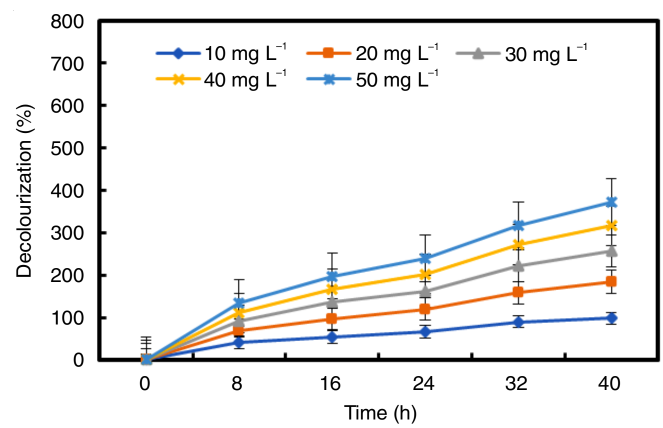

Fig. 2. Plot of decolourization potential *vs.* time at different concentration on removal of Methylene blue dye by *H. rosa-sinesis*

decreased. The variation in dye degradation of concentrations 10, 20, 40 and 50 mg L^{-1} were 86, 75, 65, 58 and 49%, respectively in case of methylene blue. Similar behaviour was reported for *C. vulgaris* with methyl red dye [23]. Further, reactions with more higher concentrations (100 mg L^{-1}) likely to be toxic for the plant. Toxicity of dye is prominent at higher concentration of dye, which negatively influence the metabolic activity of plant [21]. Therefore, at this stage no further degradation take place and the equilibrium stage is attained.

Effect of pH: Various phytoremediation experiments were greatly affected by the pH of dye solution in aqueous phase. The impact of pH on the removal of methylene blue dye using *H. rosa-sinesis* was assessed in the pH range 3 to 10 with 100 mL of 20 mg L^{-1} methylene blue dye solution. It was found that at very low and high pH value, decolourization potential decreases (Fig. 3). The dye solution pH may be influenced by the functional group interactions of dye and plant surfaces [23]. Maximum 73% decolourization of methylene blue was obtained at pH value 6. Above the value of pH 7, there was a spiky decline in the decolourization percentage. In alkaline medium, rate of removal of the dyes reduces as dye molecules competes with large number of OH^- ions. In literature, similar comparable trends of pH impact were reported in the earlier studies for remediation of methylene blue dye with help of *E. crassipes* [32]. Hence, the optimum range for the pH removal

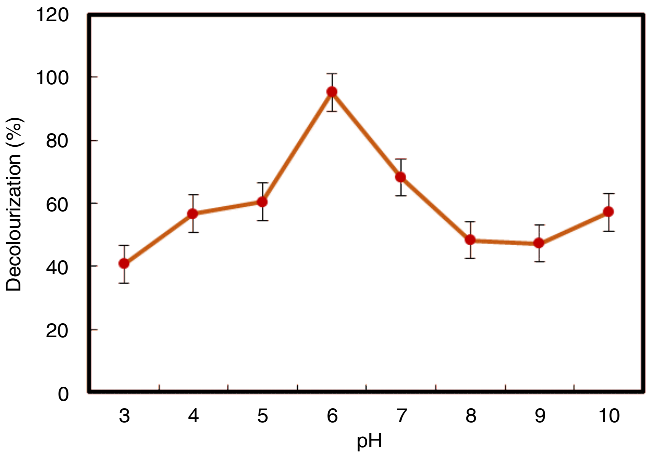

Fig. 3. Plot of impact of pH on decolourization of Methylene blue dye by *H. rosa-sinesis*

of methylene blue dye by phytoremediation of *H. rosa-sinesis* was 6.

Kinetic studies: The mass transfer mechanism is used for explaining the classical kinetic models for the phytoremediation process. The pseudo-first, pseudo-second order and Elovich equations were used to predict the kinetic study of methylene blue dye with *H. rosa-sinesis* [24,33]. The rate constant value and amount of dye removal up to equilibrium stage for different concentrations has been determined. The kinetic models of different concentrations for methylene blue dyes are shown in Fig. 4a-c. The kinetic rate constants and other parameters are determined from plots of kinetics, represented in Table-1 and concluded that R_2 values lies between (0.88 to 0.96) for methylene blue dye for 10, 20, 30, 40 and 50 mg L^{-1} concentrations in case of pseudo-first-order, which is higher than comparison to pseudo-second order (0.796 to 0.908) and Elovich model (0.694 to 0.880).

Moreover, the $q_{e(exp)}$ values for the phytoremediation of methylene blue dye by *H. rosa-sinesis* obtained were more reliable to $q_{e(exp)}$ of pseudo-first order whereas for pseudo-second order and Elovich model $q_{e(theor)}$ values were largely mismatched with $q_{e(exp)}$. Since, the standard deviation values of Δqt_1 (%) are smaller than those of Δqt_2 (%) and Δqt_3 (%), the data are more likely to be acceptable for pseudo-first order. The value of the constant has been declined with the increase in the concentration, from this it is clear that adsorption sites of methylene blue dye was less in number at the *H. rosa-sinesis* surface and the interaction of functional groups might be responsible for uptake the dye molecules.

Adsorption isotherms: The adsorption isotherm findings of methylene blue dye with *H. rosa-sinesis* are given in Table-2. The R_2 value for both isotherms were nearer to 1 *i.e.* 0.99 and 0.97 for Langmuir and Freundlich isotherms, respectively, which confirmed that the adsorption isotherm of methylene blue dye on surface of *H. rosa-sinesis* was well fitted with Langmuir isotherm. The monolayer uniform and specific at the adsorbent surface are depicted by Langmuir adsorption

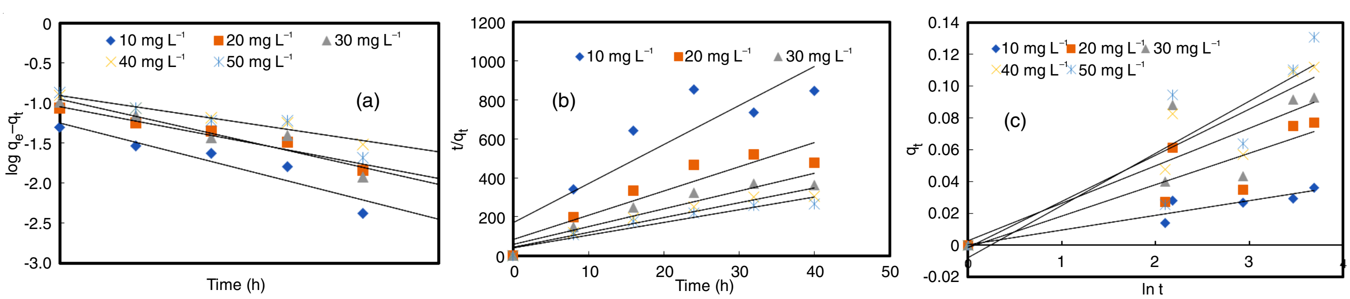

Fig. 4. Plot of (a) pseudo-first order, (b) pseudo-second order and (c) Elovich model for phytoremediation of Methylene blue dye by *H. rosa-sinesis*

TABLE-1
KINETIC PARAMETERS FOR PHYTOREMEDIATION OF METHYLENE BLUE DYE BY *H. rosa-sinesis*

Kinetic model	Kinetic parameters	Initial dye concentration (mg L^{-1})				
		10	20	30	40	50
Experimental value	$q_{e\,(exp)}$ (mg g^{-1})	0.347	0.528	0.738	0.928	1.183
Pseudo-first order	K_1	1.447	1.527	2.146	2.223	2.339
	$q_{e\,(theor)}$ (mg g^{-1})	0.328	0.587	0.788	0.866	0.996
	R_1	0.887	0.943	0.899	0.968	0.881
	Δqt_1 (%)	16.23	15.37	14.15	13.04	12.58
Pseudo-second order	$q_{e2\,(theor)}$ (mg g^{-1})	0.302	0.493	0.542	0.690	0.756
	K_2	18.6	6.26	5.14	4.13	1.54
	R_2	0.796	0.855	0.898	0.919	0.908
	Δqt_2 (%)	2.83	2.24	1.86	0.95	0.86
Elovich equation	$q_{e3\,(theor)}$ (mg g^{-1})	0.458	1.300	1.512	1.658	2.332
	α	0.005	0.007	0.012	0.013	0.015
	β	81.86	28.15	24.13	19.83	16.15
	R_3	0.880	0.758	0.694	0.840	0.750
	Δqt_3 (%)	0.512	0.452	0.356	0.267	0.157

TABLE-2
ADSORPTION ISOTHERM PARAMETERS FOR PHYTOREMEDIATION OF METHYLENE BLUE DYE BY *H. rosa-sinesis*

Langmuir isotherm constant		Freundlich isotherm constants	
Q_0 (mg g^{-1})	0.226	1/n	0.590
b (mg g^{-1})	0.086	K_F	0.0234
R_2	0.99	R_2	0.97

while heterogeneous nature of the surface effect the Freundlich adsorption model [34]. Data revealed that the surface of *H. rosa-sinesis* is a heterogeneous in nature. The value of R_L equilibrium constant decreased with the increase in concentration for 50 mg L^{-1} methylene blue dye concentration R_L value obtained 0.0198 for methylene blue dye, which suggested that methylene blue dye adsorption on the surface of *H. rosa-sinesis* is more favourable in high concentration because of interaction between adsorbate and adsorbent [29]. As the value of 1/n calculated to be 0.430 for methylene blue dye adsorption, which shows that supportive adsorption of methylene blue dye at the surface of *H. rosa-sinesis* plant since the value obtained was nearer to zero [35].

FTIR studies: The FTIR spectra employed to understand the proposed phytoremediation mechanism by interpreting the interaction between methylene blue molecules and functional groups on plants. Table-3 shows the functional groups before and after interaction of *H. rosa-sinesis* with methylene blue dye. The *H. rosa-sinesis* consists of functional groups such as O-H, C–H and C–O at the wavenumber of 3404.9, 2920.4, 2387.4 cm^{-1}, respectively and these corresponds to the cellulose, polysaccharide and carboxyl acid [24,36]. There is shifting of peak from 3404 cm^{-1} to 3466 cm^{-1} because of the O–H functional group stretching vibration [23] (Fig. 5).

TABLE-3
FTIR SPECTRAL ABSORPTION PEAKS (cm^{-1}) FOR METHYLENE BLUE DYE REMOVAL BY THE *H. rosa-sinesis*

Band	Before treatment	After treatment
–O–H (hydroxyl group stretching)	3404.0	3466.00
$-CH_2-$ (alkyne group stretching)	2920.4	2929.70
–C=stretching	2387.4	2704.23
–C–N	1153.6	1144.33
–C-O stretching	1420.1	1405.20

The change of these peak positions shows that different functional groups were involved in bonding between methylene blue dye and plant, which form complex chemical compounds [37,38]. The new peaks appearing at 2929.73, 2704.23, 2844.03 and 1144.33 cm^{-1} describe C≡C (*str.*), C=C (*str.*), C=C=C (*str.*) and feeble C–N (*str.*), respectively after the interaction of methylene blue dye with *H. rosa-sinesis*. From these interaction, it is predicted that different chemical reaction implicated chemical reaction in the phytoremediation process system [29]. Based on FTIR findings, both electrostatic and hydrogen bonding are involved during the removal of methylene blue dye using *H. rosa-sinesis* [23,24]. The main adsorption site on *H. rosa-sinesis* are carboxyl group C=O and hydroxyl group O-H at 3404.9 and 2920.4 cm^{-1}, respectively. The N^+ cationic ion present in methylene blue molecule is attracted toward hydroxyl ion (^-OH) group present in roots by H-bonding in acidic and basic conditions, which results in the adsorption of dye on root surface [29].

Comparative studies: The biosorption of methylene blue dye using modified biorenewable material as an adsorbent was recently proposed by several researchers [39]. Yet, the biosorption material is inexpensive and easily accessible, but the modification process needs either chemicals or technology. Despite all these methods, phytoremediation emerges as a practical and affordable approach for cleaning the dyes from environment to address these cost-related and technical problems [40]. In literature, only a few aquatic plants are suggested for phytoremediation of methylene blue as listed in Table-4. The only an issue with the aquatic plants is that most of these plants are not survived after treatment process and disposal of phytoremediation waste causes another challenge. Hence, in the current work a terrestrial plant *H. rosa-sinesis* was suggested for removing methylene blue dye in aquatic system hydroponically. With best to our knowledge, till date no terrestrial plant is reported for the phytoremediation of methylene blue dye. However, its decolourization percentage is little bit less in comparison to other aquatic plants (Table-4) but its survival rate and other benefits overlook this challenge. *H. rosa-sinesis* not only treat dye wastewater but also provide aesthetic look to environment and flowers for commercial use. Hence, the present study suggests that *H. rosa-sinesis* plant for synthetic dye wastewater treatment.

Conclusion

A considerable potentibility of terrestrial plant *H. rosa-sinesis* in aquatic system to remove methylene blue dye from wastewater was successfully investigated. The various para-

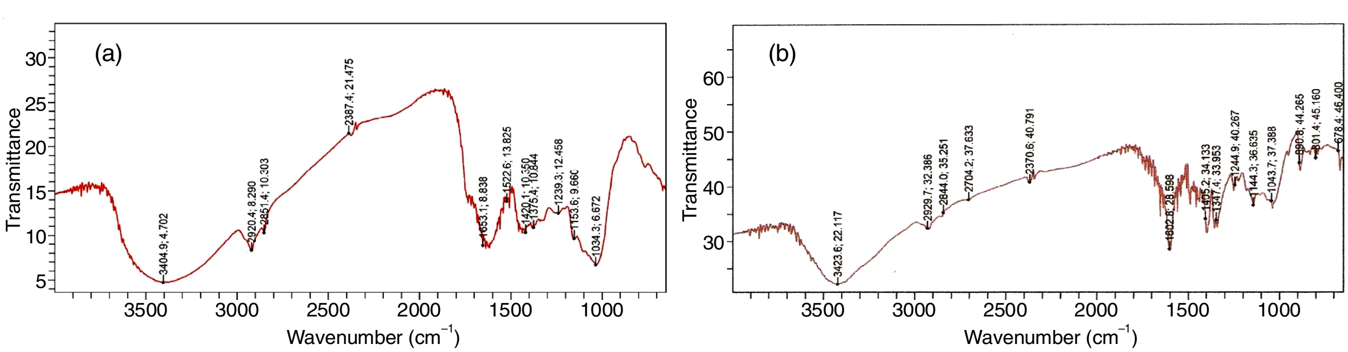

Fig. 5. FTIR spectra of *H. rosa sinesis* (a) before and (b) after methylene blue dye removal

TABLE-4
COMPARISON OF PRESENT STUDY FOR REMOVAL OF METHYLENE BLUE DYE WITH OTHER REPORTED PLANTS

Type of plant species	Plant species	Dye conc. (mg L^{-1})	Time (days)	Decolourization (%)	Ref.
Aquatic	*Spirodela polyrrhiza*	8	7	78	[41]
Aquatic	*Scirpus grossus*	200	72	86	[42]
Aquatic	*Eichhornia crappies*	50	20	98	[32]
Aquatic	*Azolla pinatta*	25	5	85	[43]
Aquatic	*Ceratophyllum demersum*	5	5	96	[44]
Aquatic	*Lemna minor*	50	2	82.48	[24,45]
Terrestrial	*H. rosa-sinesis*	10	1.6 (40 h)	86	Present study

meters like initial concentration, contact time and pH were optimized to determine the degradation potential of *H. rosa-sinesis* to remediate methylene blue dye in aqueous medium. The maximum decolourization (86% for 10 mg L^{-1}) occur at slight acidic side *i.e.* pH 6. The pseudo-first-order kinetics determined the mechanism of adsorption of methylene blue dye with $R_2 \geq 0.96$. The Langmuir model is found to be considerably better fit for the adsorption of methylene blue dye with $R_2 \geq 0.99$. On the basis of FTIR results, it was confirmed that the functional groups C–O, O–H and C–H in *H. rosa-sinesis* are interacting electrostatically with N^+ present in methylene blue during the adsorption process. In future, the more experimentation can be carried out to understand the internal mechanism of adsorption and degradation of methylene blue dye using *H. rosa-sinesis* plant.

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CONFLICT OF INTEREST

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

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