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A Novel Adsorbent: Barleria Cristata Leaves for Removal of Methylene Blue Dye

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ABSTRACT

The present study is focused on the potential use of cheap and Ecofriendly biosorbent, Barleria cristata (Koranti) leaves to remove Methylene Blue (M.B.) from its aqueous solution. It has been investigated through batch adsorption process. Aqueous solution of M.B. dye was stirred with known amount of adsorbent to determine the adsorption efficiency. The effect of various parameters viz. pH, contact time, adsorbent dose, particle size, concentration and temperature on removal of dye has been studied. The study revealed that the methylene blue dye (8 ppm) showed maximum removal at pH 8, contact time 40 min, particle size 105 µm and adsorbent dose 0.020g. Its adsorption followed pseudo second order kinetics. Thermodynamic analysis showed negative values of ΔG indicating adsorption was favourable and spontaneous, Negative value of ΔH indicate sorption process was exothermic and suggest occurrence of favourable adsorption, While positive value of ΔS indicate increase in randomness at liquid solid interface.

Keywords: Adsorption, Methylene Blue, *Barleria Cristata* leaves, Kinetics and thermodynamic.

INTRODUCTION

Huge amount of dyes are used in industries such as textiles, paper, plastic, paint, food and cosmetics industries. Most of the dyes are stable to light, difficult to degenerate, and non -biodegradable. Dye should be removed before discharging the effluent into the environment to minimise health damage and destruction of the ecosystem. Oxidation, adsorption, flocculation, precipitation and membrane technology are the four major technologies used to remove dye from waste water [1, 2]. In addition to this some other techniques like Ion exchange, reverse osmosis, ozonation etc. are being used for treating the effluent but all these techniques have some or other drawbacks. These methods are expensive and cause accumulation of concentrated sludge which becomes a major disposal problem. Among all these techniques adsorption is best technique used for purification of water effluent [3]. The process of adsorption is sludge free with clean operation and it completely removes dyes, even from the dilute solutions. Recently there is a trend to use natural bio-adsorbent for removal of dyes. Numerous researchers worked earlier on variety of adsorbents such as [4],Wool and Cotton fibre [Rasheed Khan et al 2005], Banana pitch (Namasivayam et

al 1993a 1993b), Biogas residual slurry [Namasivayam et al 1992a], Carbonized coir pitch [Namasivayam at al 2001a], Coir pitch [Namasivayam et al 2001b 2002], Hard wood [Asfour et al 1995], Mohogany sawdust rice husk [Namasivayam et al 1992b], Neem husk [Alaur et al 2010], Silk cotton hull, coconut tree saw dust [Kadirvelu et al 2003],Tuberose stick [Ahsan Habib et al 2006], Tamarind fruit shell [Papita Saha 2010]. In the present work adsorption efficiency of *Barleria cristata* leaves powder was investigated. MB is one of the most toxic industrial pollutants even at low concentrations. Therefore, they affect public health and cause many serious environmental problems. Hence, it is important to remove this dye effectively from the environment. For this purpose we have selected the bioadsorbents *Barleria cristata,* and batch adsorbent method.

MATERIALS AND METHODS

Materials: The leaves of *Barleria cristata* were collected from nearby home garden. The Methylene Blue dye (Molecular formula: $C_{16}H_{18}N_3ClS$, λ_{max}= 661nm) used in this study is obtained from fisher scientific (Qualigens fine chemicals).

Preparation of adsorbent: The leaves of *Barleria cristata* was washed under tap water to remove any dust or dirt adhered on the leaves. The leaves were dried in sunlight for 5 days and crushed, finely powered by using silica mortar and pestle. It was then passed through a sieve (105µm, 250µm, 420µm, 600µm, 710µm) to get uniform particle size. All these materials were stored in five different air tight plastic bottles.

Preparation of dye solution: A stock solution of the dye was prepared by dissolving 100 mg of dye in 1000 mL distilled water to make a stock solution of 100 mg L^{-1} The desired concentration of solution was prepared by diluting definite volume of the stock solution. For absorbance measurement a UV-VIS double beam PC scanning spectrophotometer (Shimadzu) was used. The concentration of dye was determined, before (C_0) and after (C_e) batch adsorption process, at 661nm.

Batch Adsorption process: Batch Adsorption process is used to study the removal of dyes. The various parameters viz. pH, concentration, adsorbent dose, particle size and contact time were optimized to obtain maximum removal of dye. Thermodynamic and Kinetic studies were carried out at optimised conditions.

Characterization of adsorbent: *Barleria cristata* leaves powder was characterised by various techniques. The IR spectrum of adsorbent before and after adsorption was studied using FTIR spectrophotometer (Shimadzu 8400) in the range of 4000-400cm^{-1} using KBr disk for reference. Surface morphology of adsorbent, before and after adsorption was studied by Field Emission Scanning Electron Microscopy (FET Nova nanoSEM-450).The energy dispersive spectrum (Brouker SLASH-6130) X-ray detector measures number of emitted X-ray as a function of energy. Which helps qualitative determination of the elements present in the sample.It was evaluated from spectrum of the energy as a function relative counts of the detected X-ray.

Experimental procedure: Adsorbent is dried at 333 ºk for one hour before use. 25 mL of 5 ppm solution of methylene blue was stirred for 30 minutes after addition of 0.1g adsorbent. A parameter to be optimized is varied while others are kept constant. Then Solution was centrifuged for 10 minutes and filtered through Whatman filter paper 42. Filtrate thus obtained was analysed using UV-VIS spectrophotometer and percentage removal was calculated from equation given below,

$$\% \text{ Adsorption} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad \text{..... (1)}$$

Where, C_0= Initial concentration of dye solution. C_e=Concentration of dye solution after adsorption.

RESULTS AND DISCUSSION

Adsorption of MB on bio-adsorbents *Barleria cristata* was studied by optimizing various parameters viz. pH, contact time, adsorbent dose, particle size, concentration and temperature.

Effect of pH: Variation of % removal as a function of pH is shown in fig.1

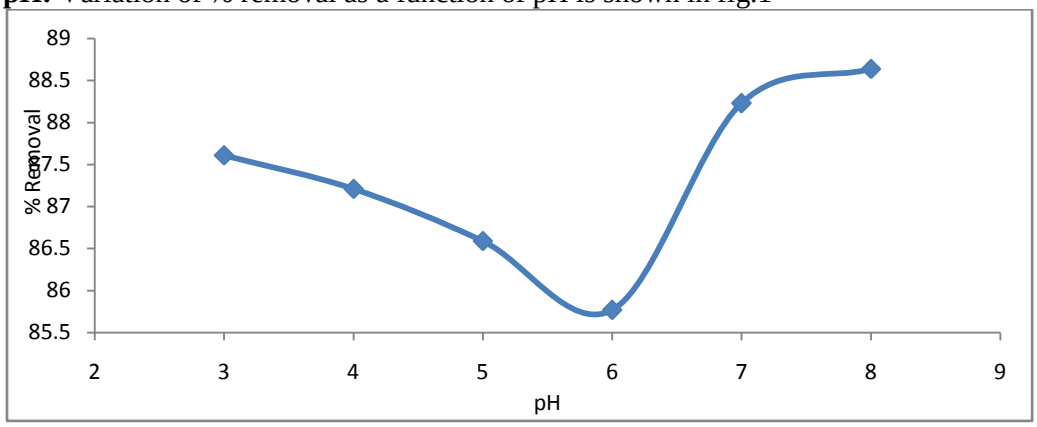

Fig. 1 Effect of pH on removal of MB [concentration 5ppm,adsorbent dose 0.1 g., contact time 30 min., particle size 420µm]

It is seen from fig.1, that the maximum removal of methylene blue (MB) is obtained at pH 8, it is also observed that adsorbed amount decreased with increase in pH up to 6 then increases up to pH 8 of the solution. The adsorption capacity increases with increase in pH of solution as methylene blue is a cationic dye. As the pH of the solution is lower, the surface of the adsorbent is positively charged and there repulsion takes place. As the pH of the solution increases, the number of negatively charge sites increased. As the initial pH became higher OH^- ions on the surface of adsorbent favour the adsorption of cationic dye due to ionic interaction between methylene blue cation and negatively charged surface of adsorbent [3]. As a result, negatively charged surface site of adsorbent favours the adsorption of methylene blue [5].

Effect of contact time: The contact time between adsorbent and adsorbate was varied between 5 to 80 min and is shown infig.2

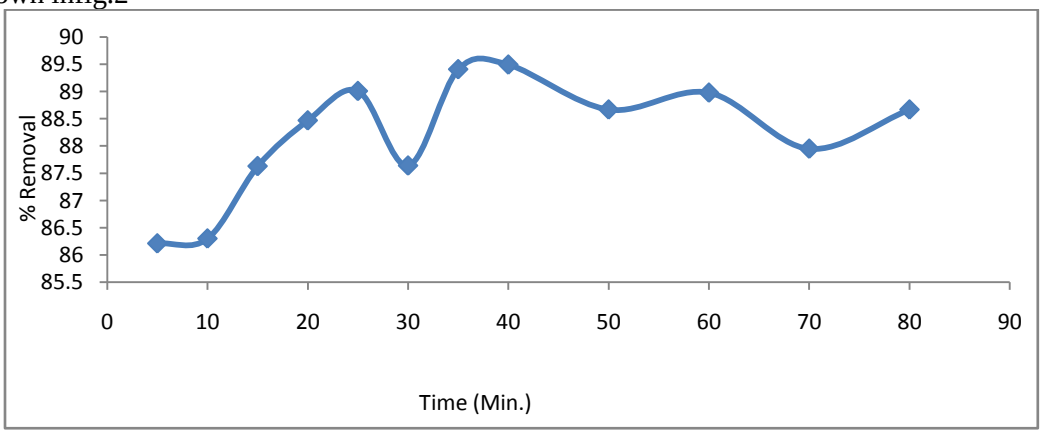

Fig.2 Effect of contact time on removal of MB [pH 8, adsorbent dose 0.1 g., particle size 420µm, concentration 5 ppm]

An examination of Fig.2 reveals that the efficiency of removal increases gradually with increase in time up to 40 minutes. Beyond this time it falls down. A somewhat similar result was reported by Rajurkaret.al [3].As contact time increases adsorption increases but at particular time interval surface area of adsorbent completely adsorbed by dye and beyond this time % removal increases that might be due to dissolution of adsorbed dyes. At this point desorbed dye is in equilibrium with adsorbed dye on adsorbent [3].

Effect of adsorbent dosage: Effect of adsorbent dose on % removal is shown in fig.3. The rate of adsorption decreases with increase in the amount of adsorbent. 0.02g. is the optimum dose for the removal of initial dye concentration of methylene blue solution.

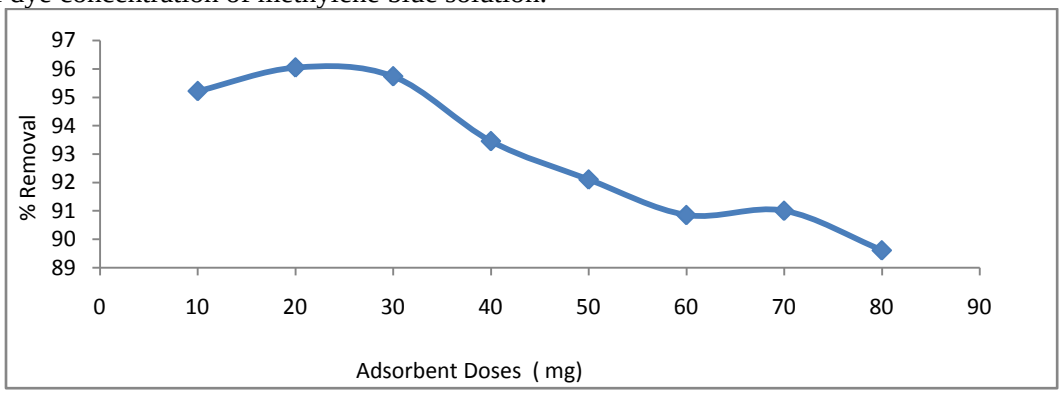

Fig.3 Effect of adsorbent doses on removal of MB [pH 8, contact time 40 min, particle size 420µm, concentration 5 ppm]

Adsorbent dose plays vital role in adsorption process by affecting adsorption efficiency of the adsorbent. The amount of dye adsorbed per unit mass of adsorbent decreases with increase in adsorbent dose [6]. Adsorption process is favoured with increased surface area and available adsorption sites. Adsorption site remain unsaturated during adsorption is most important factor. A fixed amount of adsorbent adsorbs a particular amount of dye and equilibrium attains between solute concentration in the solution and solute concentration on the surface of adsorbent.

Effect of particle size on adsorbent: Effect of particle size of adsorbent on removal of MB is shown in fig.4

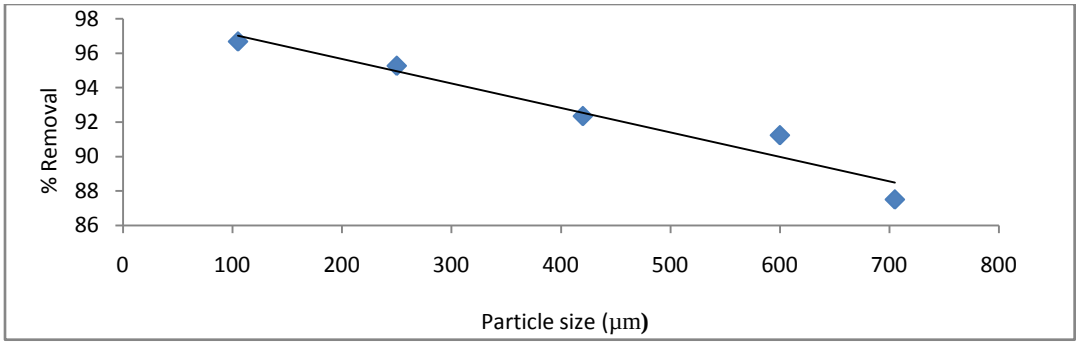

Fig.4 Effect of particle size of adsorbent on removal of MB [pH 8, contact time 40 min, adsorbent dose 0.02 g concentration 5 ppm]

As can be seen from above Figure, % removal decreases with increase in particle size and maximum dye removal was achieved at 105µm particle size of adsorbent. As particle size of adsorbent decreases, percentage removal of dye increases, this is due to increase in surface area and more availability of adsorption sites [7].

Effect of concentration on adsorbent: Variation of % removal with concentration 1 to 9 ppm is shown in Fig.5

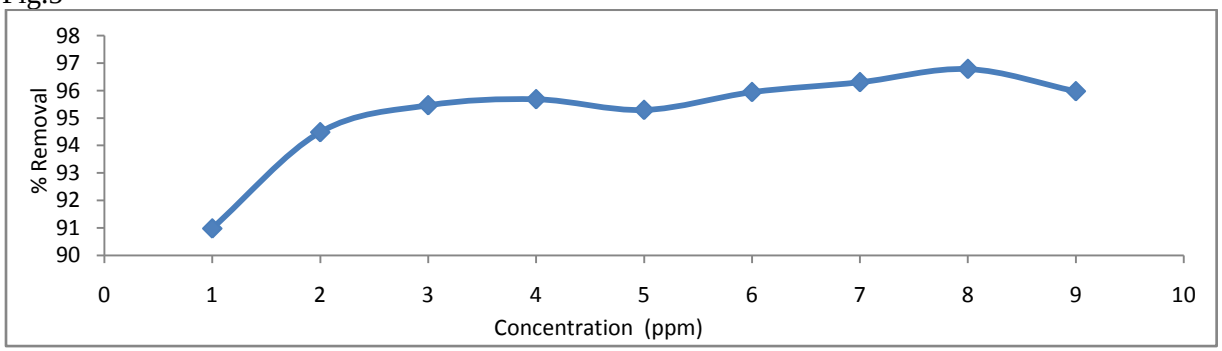

Fig.5 Effect of concentration, on removal of MB [pH 8, contact time 40 min, Particle size 105µm, adsorbent dose 0.02g]

An examination of fig.5 shows that % removal increased from 90.99 % to 96.79%, as the concentration increased from 1 ppm to 8 ppm. Amount of dye adsorbed per unit mass of adsorbent increases with increases in the dye concentration from (1-8 mg L^{-1}) at the same period of time (40 min). At 8 ppm concentration of methylene blue, the adsorption will be more due to strong driving forces of concentration gradient [8]. The total adsorption available sites at the surface of the adsorbent remain fixed for variable concentration. At this point (8 ppm), the concentration of solution and available adsorption site are at equilibrium, beyond this concentration, adsorption equilibrium and adsorption efficiency decreases due to less availability of adsorption sites.

Effect of Temperature on adsorbent: Effect of temperature, on % removal is shown in fig.6.

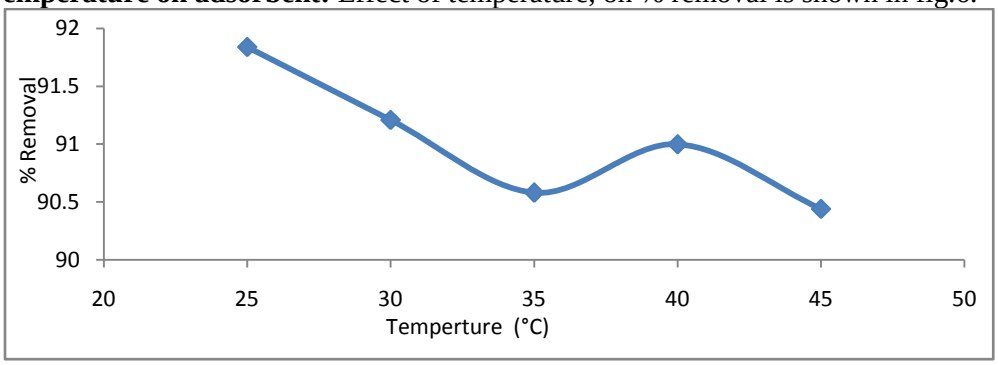

Fig.6 Effect of temperature on % removal of MB[concentration 8 ppm, pH 8, contact time 40 min, particle size 105µm, adsorbent dose 0.02 g]

From Fig.6, it is observed that the amount of dye adsorbed decreases with increase in temperature that was due to solubility of adsorbed MB increases with increase in temperature. It is concluded that 298 ºK is most favourable temperature for adsorption of methylene blue on adsorbent. At this temperature 91.84% removal was observed.

Adsorption Isotherms studies: Optimised conditions of the system are used to study the adsorption isotherms.

Freundlich isotherm: Following equation was used for the adsorption of MB onto the adsorbent as [2],

$$\log q_e = \log K_f + 1/n \log Ce \text{----------(1)}$$

Where:-q_e = Amount of MB adsorbed at equilibrium,

Ce = equilibrium concentration of MB in solution,
K_f & n = Constant incorporating factor affecting the adsorption capacity and intensity of adsorption respectively.

Good linearity is observed for a graph of log q_e verses log Ce (R^2=0.913) **(**Fig.7) which indicate that the adsorption of methylene blue obeys Freundlich adsorption isotherm with Freundlich Constant (K_f=93.54) and (n=0.5814). The adsorption intention 'n' was found to be 0.5814 i.e. not in the range 1<n<10. But higher value of K_f indicates the amount of methylene blue per unit weight of adsorbent is significantly higher [4].The higher value of K_f indicates easy adsorption of MB on adsorbent(Mahvi et al,2004) [14].

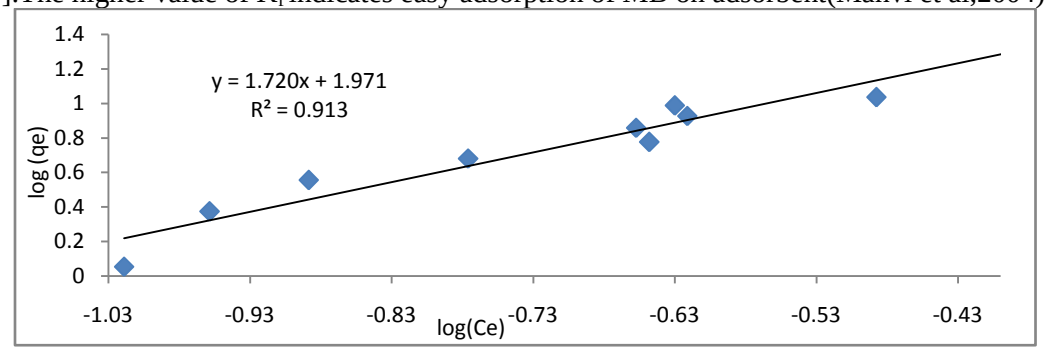

Fig.7 Freundlich isotherm plot of effect of adsorbent and dye concentration on adsorption of Methylene Blue on *Barleria cristata* at equilibrium

Langmuir Isotherm: Following equation was used for the adsorption of MB onto the adsorbent.

$1/q_e = 1/ab \times 1/Ce + 1/b$---------- (2)

Where:-q_e = Amount of MB adsorbed at equilibrium,
Ce = Equilibrium concentration of MB in solution,
a = Langmuir constant (K_L) related to adsorption efficiency (L mg^{-1}),
b= The maximum adsorption capacity (q_m) related to complete monolayer coverage (mg g^{-1})

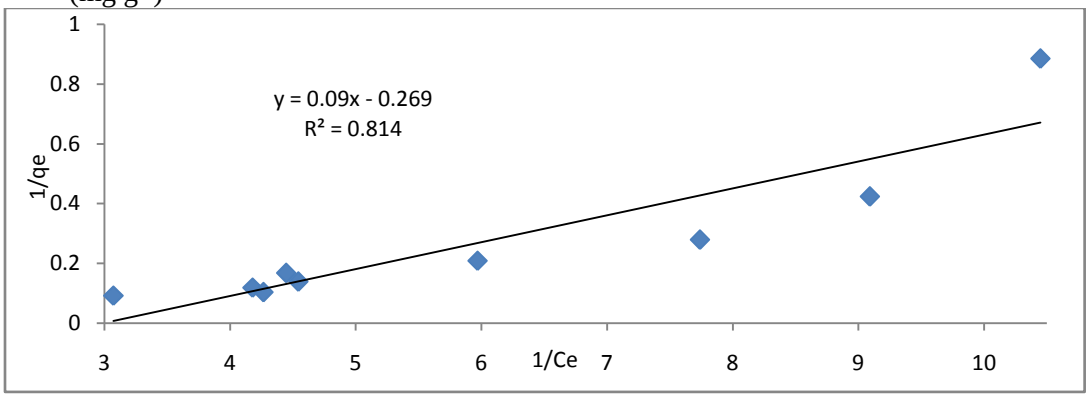

Fig.8 Langmuir isotherm plot of effect of adsorbent and initial dye concentration on adsorption of methylene Blue on *Barleria cristata* at equilibrium

Plot of $1/q_e$ verses $1/C_e$ of Langmuir adsorption isotherm ($R^2 \neq 1$), showed non-Linearity. Lower value of K_L indicate the amount of methylene blue per unit weight of adsorbent is smaller [4].Value of (K_L=a=−2.9895), (q_m=b=−3.717) and dimensional separation factor (R_L) does not lie between 0 to 1. Hence Langmuir adsorption isotherm does not fit well for adsorption of MB on studied adsorbent [8].

Adsorption Kinetic studies: A kinetic study of adsorptions provides information about its mechanism. For this purpose various kinetic models were observed.

Following equation was used for the adsorption kinetic of MB onto the adsorbent.

$$\log (q_e - q_t) = \log q_e - K_1 / (2.303)t \text{----------(3)}$$

where q_e and q_t are amount of dye adsorbed (mg g^{-1})on adsorbent at equilibrium at time respectively, K_1 is rate constant of pseudo –first order adsorption (min^{-1}).

The slope and intercept values determine pseudo –first order rate constant (k_1). Largergen pseudo –first order kinetic does not fit well ($R^2 \neq 1$) for whole range of contact time [2].
Largergen pseudo –second order rate expression is as follows [2].

$$t/q_t = 1/(K_2 qe^2) + t/q_e \text{----------(4)}$$

Where, K_2 is rate constant of second order adsorption (g mg^{-1} min^{-1}).

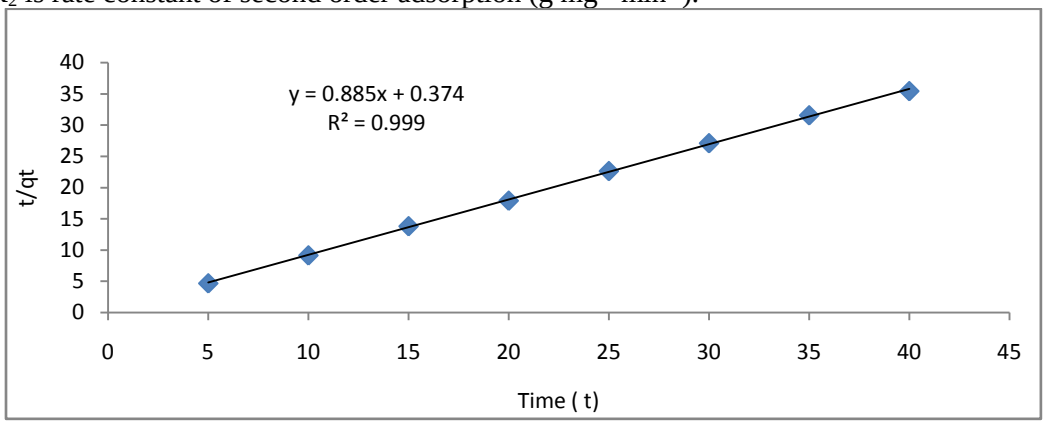

Fig.9 Pseudo –second order plot of effect of initial dye concentration and contact time on adsorption of metylene blue on *Barleria cristata.*

The plot of‘t/qt’ versus‘t’ in fig.9 was used to determine second order rate constant. Higher value of Rate constant (K_2 =2094.85) and R^2= 0.999 showed that Largergen pseudo–second order adsorption equation fit well for whole range of contact time [2]. The R^2 value indicates that the experimental results shows better fit to pseudo –second order. The equilibrium data was fitted to pseudo –second order kinetic model.

Adsorption Thermodynamic studies: The thermodynamic parameter reflects the feasibility and spontaneous nature of the process. The following equation is used to calculate various thermodynamic parameters such as change in free energy (ΔGº) kJ $molK^{-1}$, enthalpy (ΔHº)kJ mol^{-1}, and entropy (ΔSº) kJ mol K^{-1}, were used to determine at optimised conditions.

$$\Delta G^o = \Delta H^o - T\,\Delta S^o \text{---------- (5)}$$

$$\Delta G^o = -RT\ln K_0 \text{---------- (6)}$$

$$-RT\ln K = \Delta H^o - T\,\Delta S^o$$

$$\ln K = \Delta S^o/R - \Delta H^o/RT \text{ ---------- (7)}$$

Where: K is equilibrium constant, R is gas constant, T is temperature in ºK, ΔGº, ΔSº and ΔHº can be calculated by van’t Hoff equation (5). ΔGº is calculated from equation (6), ΔHº and ΔSº value obtained from the slope and intercept of plot lnK verses 1/T, The negative value of ΔGº indicates the adsorption is spontaneous and thermodynamically favourable adsorption for methylene blue. The very low negative value of ΔHº indicates physisorption and reaction between dye and adsorbent is endothermic nature of adsorption [2]. The positive value of ΔSº indicates change in surface morphology and increase in randomness at solid liquid interface [9].

A plot of ln K verses 1/T is thermodynamic studies of *Barleria cristata* adsorbent

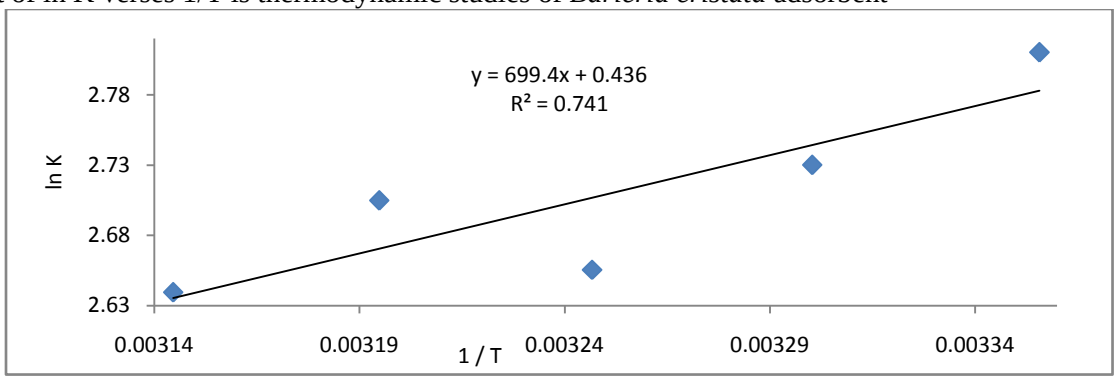

Fig.10 Plot of ln K Vs 1 /T for methylene blue

Thermodynamic parameters obtained from these plots are recorded in following table 1.

Table :1 Thermodynamic parameter for methylene blue sorption by *Barleria cristata* adsorbent

Sr. No.	Temp. (ºK)	ΔGº(kJ/mol K)	ΔHº(kJ/mol)	ΔSº(kJ/mol K)
1.	298	−6962.47	−5814.81	3.625
2.	303	−6877.51		
3.	308	−6799.97		
4.	313	−7038.65		
5.	318	−6978.45		

As the temperature increases from 25ºC to 45ºC the percentage removal of methylene blue dye decreases from 91.84% to 90.44% because as temperature increases ,the diffusion rate of adsorbate molecule across the external boundary layer and interval pores of the adsorbent particles decreases to some extent. Temperature change will change the equilibrium capacity of the adsorbent for particular adsorbate [5]. From above table, the ΔGº values are negative indicating the spontaneous nature and thermodynamically favourable adsorption for methylene blue [9]. The negative value of ΔHº indicates exothermic process [10]. The positive value of ΔSº indicates increase in randomness at liquid solid interface [9].

Adsorption Characteristics

FTIR Studies: The Spectra of virgin and methylene blue loaded adsorbent are shown in fig.11.

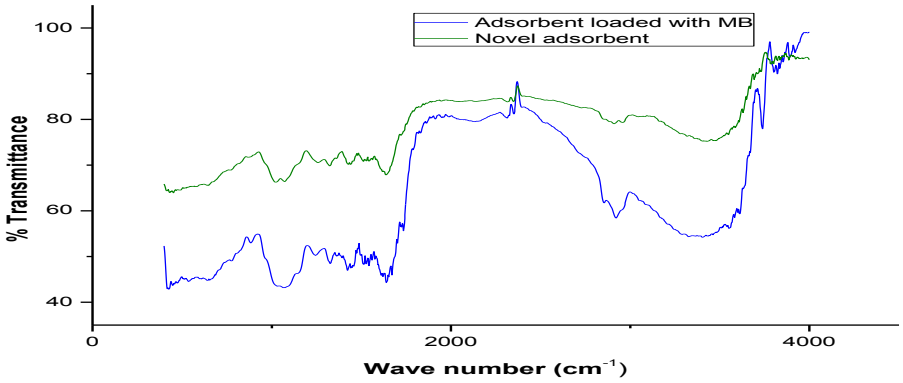

Fig. 11 FTIR Spectrum of *Barleria cristata* leaves powder (a) and MB loaded *Barleria cristata* leaves powder (b).

I.R. absorption spectra of the *Barleria cristata* (a) and organic dye Methylene blue adsorbed on *Barleria cristata* (b)is shown in Fig.11. FTIR spectrum of novel adsorbent (a) shows a peak at 3358–3318 cm^{-1}which indicates the presence of H –bonded –OH stretching vibration in phenolic compound present in cellulose of adsorbents .In spectrum (b), Strong broad band at 3610–3738 cm^{-1}due to -OH stretching vibration, peak at 289 cm^{-1}, 3325 cm^{-1}, 3381 cm^{-1} indicates the presence –NH stretching vibration and band at 3051–3086 cm^{-1} is due to –OH stretching vibration in ring. The absorption band at 2854 –2920 cm^{-1} can be assigned to symmetric and asymmetric stretching vibration of the C–H_3 bonds in the dimethylene amino groups of methylene blue molecule [11]. A sharp band at 1244–1325 cm^{-1} is due to –C–N stretching in aromatic amines. The band at 1423–1454 cm^{-1} was due to C–C stretching in ring. The band at 773–881 cm^{-1} is C–H bending vibration in heterocycle [11]. The sharp band at 1638–1670 cm^{-1} is due to C=C bending vibration in heterocycle. The band at 3610–3738 cm^{-1}, 3211–3246 cm^{-1}, 1670–1734 cm^{-1}, 1454–1494 $cm^{-1}$are not seen in spectrum (a).It is seen from figure, the peak intensity of spectrum (a) and (b) was changed considerably. The change in intensity indicates methylene blue is adsorbed on to the adsorbent by adsorption process.

FESEM Studies: The morphological characteristics of adsorbent were studied using field emission scanning electron microscope.
The FESEM micrograph are shown in fig.12

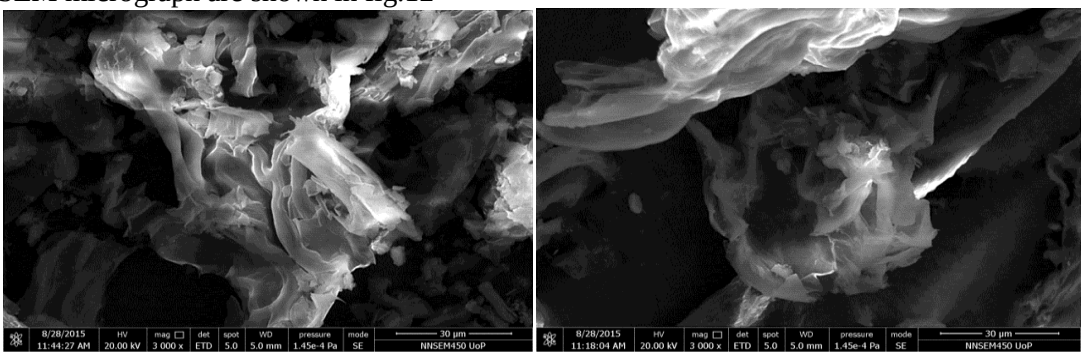

Fig.12a SEM micrograph of *Barleria Cristata* **Fig. 12b** *Barleria acristata* loaded with MB

SEM micrograph of virgin adsorbent, shows irregular, rough and highly porous surface indicating the possibility of good adsorption. The appearance of a molecular cloud (darkness) over the surface of the dye loaded adsorbent (Fig.12b) confirms the adsorption of dye on to the surface of adsorbent. There are no porous (void) spaces on the surface of adsorbent because 91.84% dye adsorbent on to the surface of adsorbent.

EDX studies: The EDX spectrum of *Barleria cristata* leaves powder and MB loaded *Barleria cristata* leaves powder is shown in fig. 13a & 13b respectively.

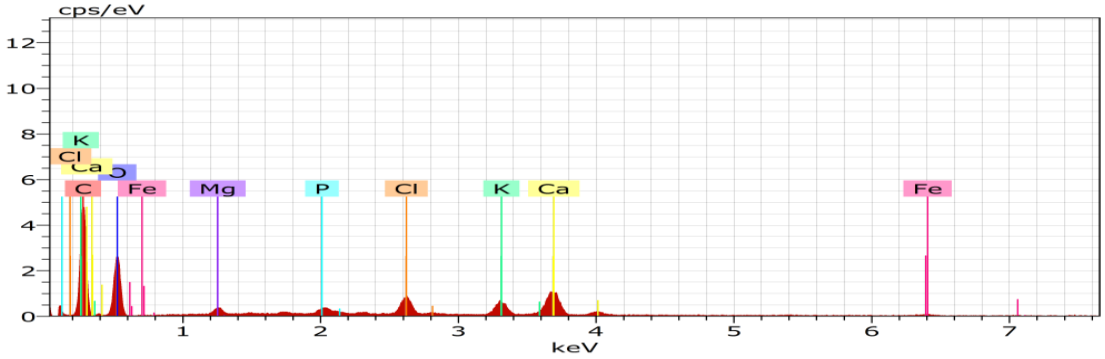

Fig. 13a EDS of *Barleria cristata*

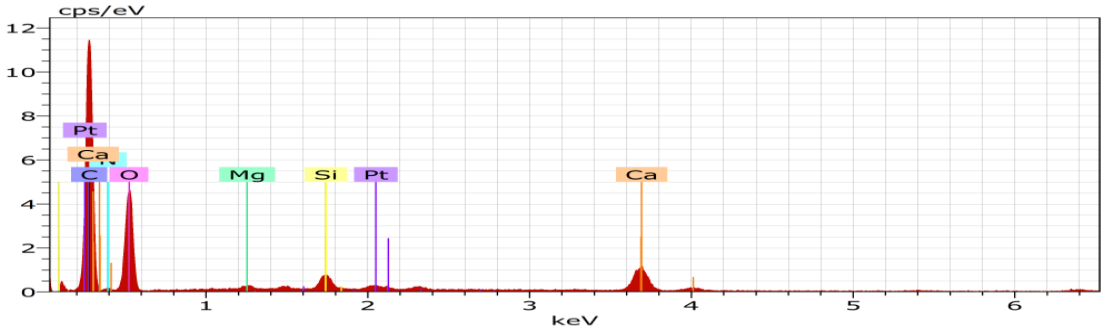

Fig. 13b EDS of *Barleria cristata* loaded with MB

Energy dispersive spectrum of adsorbent (13a) shows the elements C, O, Ca, K, Cl, Mg, P, Fe. While Energy dispersive spectrum of adsorbent loaded with methylene blue (13b), shows additional element 'N'. From figure, it was concluded that adsorbent loaded with methylene blue shows presence of 'N' which was present in the dye.

APPLICATIONS

Barleria cristata leaves powder can be used for effective removal of methylene blue from aqueous solution. The technique is economical and efficient, novel adsorbent is easily available and can be applied to other dyes also.

CONCLUSIONS

From the data of the present study, it is concluded that, the adsorption process is a very effective method for removal of methylene blue from aqueous solution.

• Maximum removal (91.84%) of Methylene blue (8 ppm) was observed at pH 8, contact time 40 min, adsorbent dose of 0.020g, particle size of 105 µm.

• Thermodynamic study revealed that adsorption of methylene blue dye is a spontaneous and exothermic process.

• Adsorption of methylene blue follows pseudo –second order kinetics.

•Untreated powdered leaves of *Barleria cristata* act as an effective adsorbent for removal of Methylene Blue dye.

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