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A Parametric Study for Adsorptive Removal of Methylene Blue using Low-Cost Adsorbent

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Abstract:

This research work is focused on removal of methylene blue dye using carbonaceous material produces from Spanish Cherry fruit peel as adsorbent. The effect of various parameters such as temperature ofadsorbent preparation, contact time, initial dye concentration, adsorbent doses, pH of the dye solution and operating temperature on percent removal of dye was investigated.It was found that maximum 81.5% removal of dye was obtained for adsorbent dose 1 gm/lit (carbonization temperature 650^{o}C) and pH of the solution 7 at 30^{o}C for 1 hour under the range of parameter studied.The experimental data was verified with Langmuir, Freundlich and Temkin isotherm model. Although the correlation coefficient(R^2) values imply that the three models are applicable for this adsorption process.It is observed that Temkin model best agreed with experimental results. Maximum adsorption capacity for monolayer adsorption from Langmuir adsorption isotherm study was found 77.52 mg/gm. The results suggest that the Spanish Cherry fruit peel can be used as an efficientadsorbent for Methylene Blue removal from waste water.

Key Words:Adsorption,Dye removal, Low-cost adsorbent,Methylene Blue, Spanish Cherry fruit Peel

1. INTRODUCTION:

The concern of save water and save life becomes more emphasizing in recent days because of rising level of water pollution.The effluent coming from different industries carries various types of organic and inorganic contaminants whichare responsible to pollute waterbodies [1].More than 10000 tones dyes per year have been used during the dying process, which is one of the important steps for textile, paper, cosmetics, pharmaceutical, leather and rubber manufacturing industries [2][3]. Most of the dyes are synthetic in nature which carries at least one colour bearing group (chromophores) and colour helper (auxochromes) which are highly toxic, as well as their complex chemical structure categorized them as non-biodegradable and carcinogenic to living beings [4][5]. In textile and dye industries, 10-20% dyes are lost during the dying process and up to 2 to 20% of the dyes cannot bind to the material and are discharged directly into the water stream, making the water-body exceedingly coloured and aesthetically unpleasant for marine life [6]. Due to this reason the demand of dye removal process increasing day by day to improve our ecosystem.

This study is focused on methylene blue dye removal from waste water. Methylene blue is a cationic dye which is basic in nature [7]. It is mostly used in textile, leather and paper industries [8]. This dye causes serious health hazards by affecting central nervous system with symptoms of dizziness, mental confusion, headache. It causes irritation in digestive system leads to nausea, vomiting, abdominal pain and also causes irritation to eyes,skin and in renal system. Intake of this contaminants can also disturb the respiratory system by reducing the red blood cell count and haemoglobin[5].

Differentefficient techniques such as chemical oxidation, chemical precipitation, electrochemical treatment, adsorption process, biological treatment and membrane filtration can be emplyedfor dye removal. Chemical oxidation is a rapid decolouration process but the compounds that generated as by-products are highly toxic which causes health hazards [9]. In spite of gaining high dye removal efficiency, the chemical precipitation process is not favourable due its high chemical cost [10][11][12]. Bioreactor treatment are eco-friendly and inexpensive but it is a long-timeprocess [13]. Membrane filtration is an advance technology which gives higher efficiency, but the major drawback of this process is membrane fouling. Cleaning of the membrane and treatment of the sludge generated during this process is a big challenge [13]. Another efficient process for dye removal is electrochemical [14][15][16]. But the major drawback of this process is high energy consumption and production oflarge volume of sludge [17].

Adsorption process is another alternative process for dye removal. Broad range of adsorbents including commercial, low-cost green adsorbent and nanoparticles has been used for dye removal [18][19][20][21][22]. Among all process, adsorption is most emphasizing process as the process is simple, flexible and economically feasible [6] [4][23]. Use of nanoparticles are also becomes hurdle for its toxic disposal [13]. In recent years, the commercial adsorbents are replaced by low-cost bio-adsorbents to make the process cheaper and more sustainable [24]. Various agricultural waste such as ground nut shell [25], pumpkin peel [26], banana peel [27], cucumber peel [28], tomato peel [29], dragon fruit peel [30] etc and forest waste such as oak waste [31], artichoke leaves [32], willow bark [33] etc have been widely used for elimination of methylene blue from coloured waste water.

Spanish cherry (MinusopsElengi) is an evergreen tree mostly found in south Asian country. This tree bears fruit in between June to November. The ripe fruits of this tree are yellow coloured, fleshy and contain brown seeds inside. Abundant availability of this tree and sufficient amount of waste fruits have drawnour attention to prepare the low-cost adsorbent for waste water treatment.In this study, methylene blue has been removed from waste water by using the fruit pulp of this tree by simply converting it to carbonaceous matter instead of activated carbon.

This research work is focused on the influence of various process parameters on adsorption phenomena. The Langmuir and Freundlich adsorption isotherm study have been carried out to determine the maximum adsorption capacity.

MATERIALS AND METHODS:

Adsorbent:

The ripe fruits are collected from campus of Haldia Institute of Technology, Haldia, West Bengal, India in the month of October. Thoroughly washing of the fruits have been done by clean water to remove dirt and impurities. Washed fruits were then dipped into the Hydrochloric acid (HCl) (5 N) (Fisher Scientific India Pvt.Ltd., Mumbai-400076)solution for 24 hours to remove colour. After, one day again it has been washed with distilled water thoroughly. The fruits are then kept for 7 days under the sun for drying. Now, the seeds were easily separated from the pulp. Dried pulps have been converted to carbonaceous material in muffle furnace at 450^{o}C,550^{o}C and 650^{o}C for 15 minutes, respectively. The materials then crushed and sieved by 125µm screen. These carbonaceous materials(-125µm) of Spanish Cherry fruit peelhave been used as an adsorbent in this study. The moisture content of the adsorbent was found 9.22%.

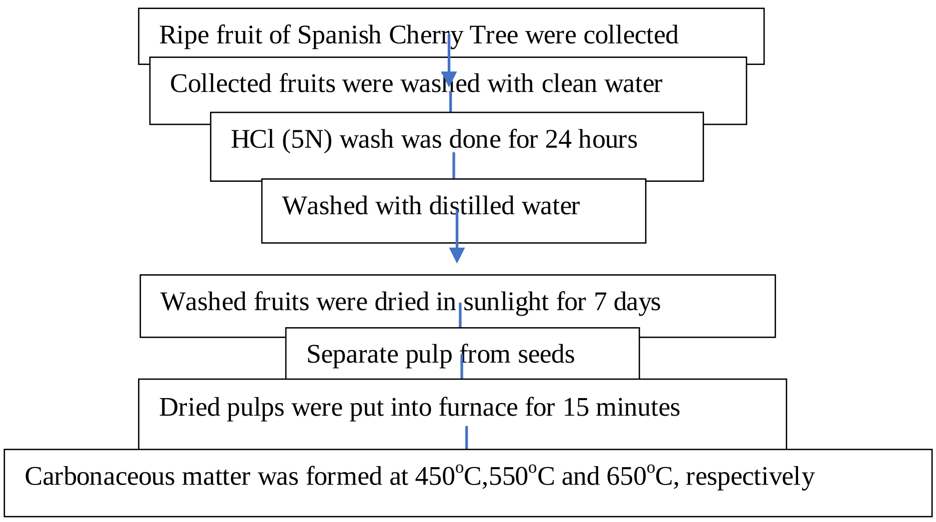

Fig. 1: Preparation of adsorbent from ripe fruit peel of Spanish Cherry tree.

Adsorbate (Dye):

Meyhtyleneblue (MB) dye isprocured from Merck Specialities Pvt. Ltd (Mumbai, 40018). It is a mono valent cationic dye. The properties of the dye are given in table 1.A 1000 gm/lit stock solution have been prepared by using distilled water. Successive dilution of the stock solution has been done for preparing the solution of differentconcentrations of 50-200 ppm,used in experimental procedure.

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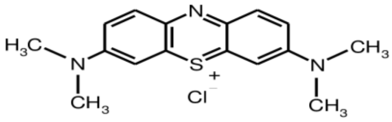
Properties of Methylene Blue	
Common Name	Methylene Blue
IUPAC Name	3,7-bis (Dimethylamino) phenazathionium chloride
Other Names	Basic Blue 9, Tetramethylthionine chloride
Molecular Formula	$C_{16}H_{18}CIN_3S$
Molecular Weight	319.85 gm/mole
Maximum Wavelength, λ_{max}	664 nm
Chemical Structure	

Table 1: Properties of Methylene blue.

Adsorption study:

Series of experiments have been carried out to investigate the influence of process parameterson adsorption of MB dye using prepared bio-adsorbent. In a single system, adsorbent (preparing temperature range from 450°C-650°C) dose ranges from 0.5-2.5gm/lit was added into a 100 ml dye solution of concentration range from 50-200ppm in a conical flask.The conical flask was then kept on the hot plate stirrer with an agitation speed of 60 rpm adjusting the temperature range from 25°C to 65°C for each run.The solution was withdrawn at a fixed time interval to measure the residual concentration by using UV-Spectrophotometer by setting the wavelength at 664nm. The adsorption process was carried out for 1 hour.

Percent of dye removal (% R) was calculated by using the given equation;

$$\%R = \frac{C_0 - C_t}{C_0} \times 100\ \% \qquad (1)$$

The amount of dye adsorbed at time t (q_t) and at equilibrium (q_e) can be described by the given equation;

$$q_t = \frac{(C_0 - C_t) \times V}{W} \qquad (2)$$

$$q_e = \frac{(C_0 - C_e) \times V}{W} \qquad (3)$$

Where, $C_0$is the initial dye concentration (mg/lit) and C_t is the dye concentration (mg/lit) at time t, V is the volume of the dye solution (L) and W is the mass of adsorbent (gm).

Adsorption isotherm study:

Langmuir, Freundlich isotherm model were used to verify the experimental data. The correlation coefficient values (R^2) closer to unity decide the best fitted isotherm model.

Langmuir adsorption isotherm was developed based on homogeneous sites on adsorbent surface. At the point of adsorption saturation, the monolayer was formed by adsorbate on the adsorbent surface. The linearize form of Langmuir Adsorption isotherm [34] can be described as:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \qquad (4)$$

Where, K_L is the Langmuir constant related to the binding affinity between adsorbent surface and adsorbate in (L/mg) and q_m is the monolayer adsorption capacity in mg/gm.

A dimensionless parameter known as separation factor (R_L) [35] can be expressed by Langmuir adsorption isotherm model. R_L is defined as:

$$R_L = \frac{1}{1 + K_L\ C_o} \qquad (5)$$

The nature of adsorption can be enlightened by the value of R_L.

Values of R_L	**Nature of Adsorbent**
>1	Unfavourable
=1	Linear
$0<R_L<1$	Favourable
=0	Irreversible

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Table 2: different values of separation factor (R_L)

Freundlich isotherm model was proposed based on heterogeneous sites on the adsorbent surface. The logarithmic form of Freundlich isotherm [35] can be describes as:

$$ln q_e = \frac{1}{n} ln C_e + ln K_F \quad (6)$$

Where, K_F is the Freundlich adsorption constant related to adsorption capacity ($mg^{1-1/n}L^{1/n}gm^{-1}$) and n is the dimensionless parameter related to adsorption intensity.

Temkin model was proposed based on attraction affinity between the adsorbate and adsorbent molecules. In this model it was stated that the heat of adsorption linearly decreases with the surface coverage of adsorbent by adsorbate. The equation can be stated as [35]:

$$q_e = B ln C_e + B ln K_T \quad (7)$$

Where, Kt is the equilibrium binding constant in L/gm and B=RT/b, where, b is related to heat of adsorption in J/Mole.

RESULTS AND DISCUSSION:

Effect of adsorbent preparation temperature and contact time on dye removal:

The adsorption study was done by using 1 gm/lit adsorbents which were prepared at 650°C,550°C and 450°Cin 100 ppm dye solution at 30°C. The results show (fig.-2) that the adsorbent prepared at 650°C have higher dye removing ability. The results confirm that the complete carbonization of the fruit peel was done at 650°C, below this temperature some non-carbonaceous materials were present in adsorbent which reduce the adsorption efficiency. Further, studies have been done by taking the adsorbent prepared at 650°C.

The dependence of adsorption on contact time (fig.-2) was studied and it is observed that initially the adsorption rate was very high but as the time goes on the rate becomes nearly constant. The constant rate indicates that the adsorption process achieves its equilibrium condition.

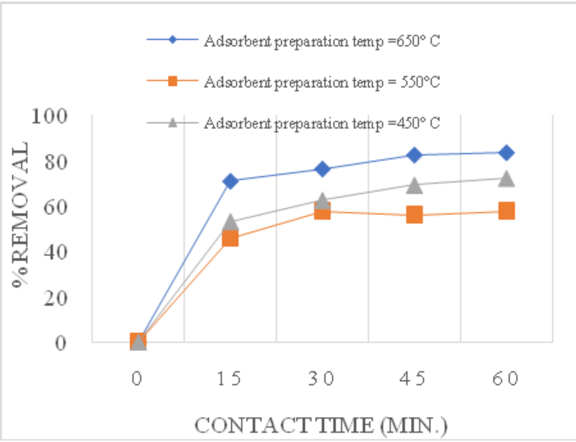

Fig.-2: Effect of adsorbent preparation temperature and contact time on dye removal

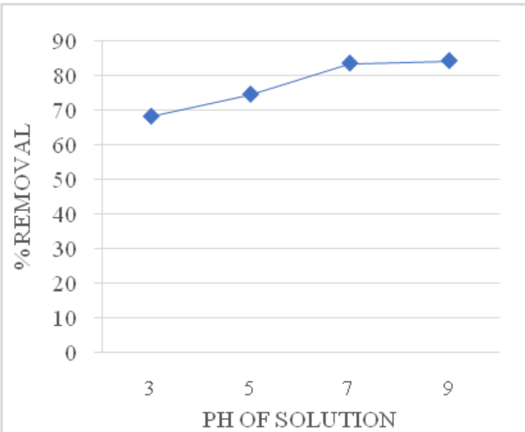

Fig.-3: Effect of pH for dye removal

Effect of pH for dye removal:

Dependency of pH on adsorption is given in fig.-3. The dye uptake was increased with increase initial pH of the solution. The % removal was found nearly same at pH 7 and pH 9. So, the optimum pH value for adsorption was be found 7 to 9. At acidic pH the solution has excess H^+ ions which compete with dye cations for occupying the adsorbent sites. Whereas, at higher pH as the number of positive ions were less, the dye cations were adsorbed very quickly on the adsorbent surface.

Effect of Adsorbent dose for dye removal:

The effect of adsorbent amount on adsorption was studied for 100 ppm dye solution at 7 pH at 30°C varying the adsorbent dose from 0.5-2.5 gm/lit. Figure 4 represents that the % removal has been increased as the adsorbent dose was increased from 0.5 to 1 gm/liter, whereas, beyond 1gm/liter no significant change have been observed. The adsorption saturation was found above 1 gm/lit. Therefore, the optimum dose was taken 1gm/lit for further studies. Initially, increased surface area confirms the increased %removal, whereas, the surface area might reduce due to the formation of agglomeration as the adsorbent dose was increased beyond 1 gm/lit.

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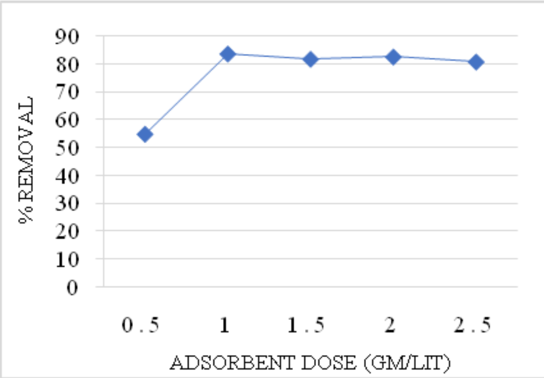


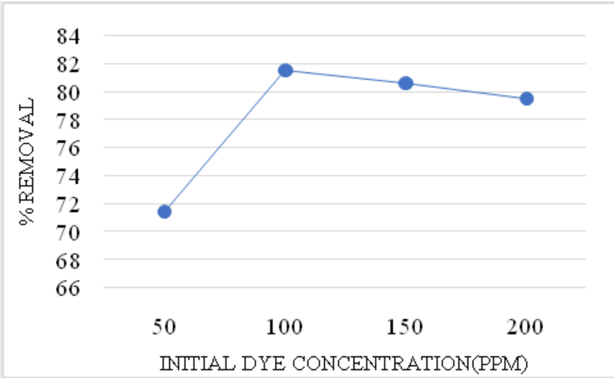


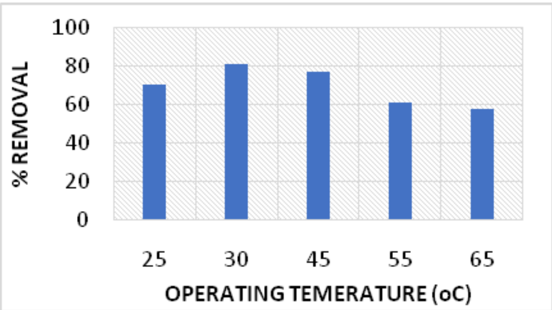
Fig.-4: Effect of Adsorbent dose for dye removalFig.-5: Effect of initial dye concentration on dye uptake

Effect of initial dye concentration on dye uptake:

The influence of the initial dye concentration on adsorption was studied by fixing the adsorbent dose 1gm/lit, pH of the solution 7 at 30°C. This phenomenon is represented by the figure-5. The dye uptake was increased as the dye concertation was increased from 50-100 ppm. Thereafter, the adsorption process was in decreasing in nature. The reason can be explained by the availability of active sites present on the surface. It might be assumed that at 100 ppm the adsorption process gets saturated afterward no vacant sites are available for adsorption of dye.

Effect of Operating Temperature on dye uptake:

The dependency of temperature on adsorption is shown in figure 6. The optimum temperature was found 30°C. Above 30°C the dye adsorption was decreasing with the increasing temperature. The reason can be explained as the temperature was increased, there was a high tendency to escape of the adsorbed molecules from the active sites of the adsorbent surface.



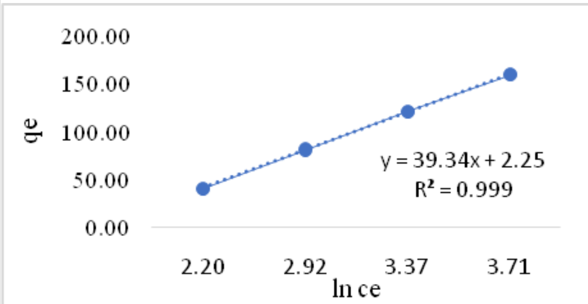


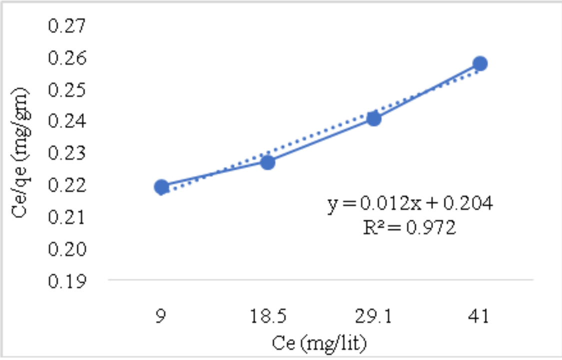
Fig.-6: Effect of Operating Temperature on dye uptake Fig.-7: Temkin adsorption isotherm model

Absorption Isotherm:

The experimental data was fitted with Langmuir, Freundlich and Temkin adsorption isotherm in figure-7 and figure-8. The adsorption parameters and correlation coefficient (R^2) were determined for these isotherm model. The values are given table-3

Langmuir Isotherm				Freundlich Isotherm			Temkin Isotherm		
q_m(mg/gm)	K_L (L/mg)	R^2	R_L	K_F $mg^{1-1/n}L^{1/n}gm^{-1}$	n	R^2	K_T (L/gm)	b (J/Mole)	R^2
77.52	0.0632	0.9721	0.158	29.35	2.24	0.9575	1.06	64.035	0.9998

Table-3: values of adsorption parameter and correlation coefficient



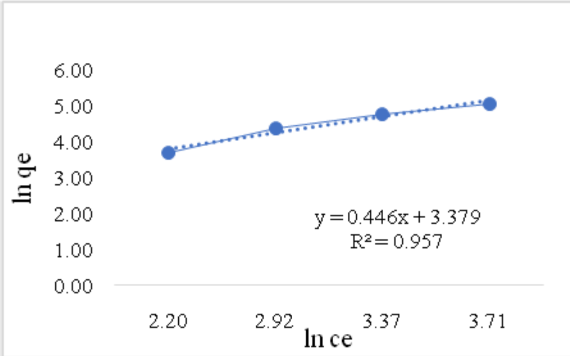


Fig.-7: Langmuir adsorption isotherm model Fig.-7: Freundlich adsorption isotherm model

From the values of correlation coefficient (R^2) (table-4) it can be said that three isotherm model can be fitted with the experimental data [36]. However, the value of R^2(0.9998) indicates that Temkin isotherm is best fitted model for our experimental data. The higher b value represents a uniform distribution of binding energy on the adsorbent surface which leads to increase the adsorption capability. Moreover, R_L value (0.158) depict that our adsorption is favorable in nature.

CONCLUSION:

In this research work, the adsorption efficiency of an adsorbent, prepared from Spanish Cherry fruit peel was investigated for methylene blue dye removal. The investigation was carried out by varying different process parameters like, adsorbent preparation temperature, adsorbent dose, initial dye concentration, pH of the solution, contact time and operating temperature. The maximum dye uptake was found 81.5% by fixing the adsorbent dose 1 gm/lit (carbonization temperature 650°C) and pH of the solution 7 at 30°C for 1 hour. The experimental data were well fitted with Temkin adsorption isotherm model. The maximum adsorption capacity was found from Langmuir isotherm 77.52 mg/gm. Results, represents that this adsorbent is good enough in terms of cost as well as removal efficiency.

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