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Kinetics and Isotherms Studies of methylene blue Basic Dye from Aqueous Solution onto *Selenicereus grandiflorus* Activated Carbon

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Asian Journal of Materials Chemistry

Volume: 4 Year: 2019
Issue: 1–2 Month: January–June
pp: 1–6
DOI: <https://doi.org/10.14233/ajmc.2019.AJMC-P71>

Received: 7 February 2019
Accepted: 29 March 2019
Published: 29 June 2019

ABSTRACT

The present experiment explains the effectiveness of adsorption studies of methylene blue dye from aqueous solutions on activated carbon from *Selenicereus grandiflorus* (SG) treated with conc. sulfuric acid. The sulphuric acid-treated *Selenicereus grandiflorus* activated carbon (SGAC) was used as low-cost adsorbent for the removal of methylene blue dye from aqueous solution. It suggests an ideal alternative method to adsorption of dye compared to other expensive treatment options. The adsorption studies have been conducted at different experimental parameters, *i.e.*, pH, contact time, adsorbent dose and initial dye concentration. The batch mode experiments were conducted by different adsorbent dose (0.03-0.150 g per 50 mL), pH of the solution (2-12), effect of time (3-18 min), initial dye concentration (10 mg/L), point of zero charge and regeneration of spent adsorbent studies. Langmuir model shows better fit to the equilibrium data ($R^2 = 0.966$) than Freundlich model. The adsorption capacity (Q_m) of SGAC increases with increasing dosage where Q_m is 16.17 mg g⁻¹.

KEYWORDS

Point of zero charge, Isotherms, Kinetics, *Selenicereus grandiflorus*.

INTRODUCTION

Increases the life passion of the people and modern civilization [1,2] effects the entire population and its growth [3], Industries discharge the contaminated, coloured waste water into water systems [4]. Organic dye pigments present in wastewater result in visual colouration even after slight colour discharge into the environment water body, which cause anxiety for public health and environmental establishment [5]. There are more than 700 tons of dyes per every years, such types of industries are textile, leather, paper, cosmetics, printing and plastics [6]. Methylene blue is used as an adsorbate for investigation, which is water soluble and mainly used in industrial activities, such as dyeing of textiles and leather, printing calico, printing cotton and biological staining methods [7], methylene blue dye is dangerous because of its no biodegradable, hazardous and toxic carcinogenic effects to all living things [8]. It causes diseases such as skin allergies like skin irritations, dermatitis and liver, brain, reproductive, central nervous system problems [9]. Basic dyes present an obvious colouration even

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Available online at: <http://ajmc.asianpubs.org>

at low concentration (< 1 mg/L) and have been classified as toxic colourants. In all developed countries assigned some scientist and research scholars for treatment of pollutants such as dyes, heavy metals and other organic impurities in all industries and sewage treatment plants in cities [10,11]. These problems are the real challenges to the younger generation and necessary to have treatment of wastewater to minimize the pollutants from the industries and discharge low/less concentrated polluted dye solutions to the water bodies.

The wastewater treatment techniques are ion exchange, chemical precipitation, membrane filtration, electrochemical technologies, adsorption and photo catalysis [12-15]. Adsorption has been found to present advantages among all other techniques for treating dye effluents or coloured wastewater because its simple operational conditions, easily available, economically good and mainly it is non-toxic substances [16-19].

The present work explains the batch adsorption method which minimizing the secondary waste because recycling is also one parameter in this method [20]. Highlights of this paper is adsorption of dye from aqueous solution on to a new fast and efficient adsorbent is *Selenicereus grandiflorus* flowers activated carbon by the batch adsorption technique. The batch adsorption studies, such as pH effect, dosage effect and effect of contact time and also explains the point of zero charge, regeneration studies adsorption isotherms, kinetic studies by using *Selenicereus grandiflorus* activated carbon (SGAC) as an adsorbent and methylene blue as the dye.

EXPERIMENTAL

Methylene blue were used as an hazardous pollutant, purchased from Merck Chemicals (India). Sodium hydroxide, hydrochloric acid, sulfuric acid were purchased from Sigma Aldrich Pvt. Ltd. (India). Double-distilled water was utilized throughout the experiment and all experimental studies triplicate.

Preparation and characterization of SGAC: White coloured *Selenicereus grandiflorus* flowers (Brahma kamala) were collected around local areas of Bangalore dried under sun light for 2 days and hot air oven for 2-3 days. The dried *Selenicereus grandiflorus* flower was ground into powder. Then it was soaked with conc. sulfuric acid and kept at $200\text{ }^{\circ}\text{C}$

for 24 h. The carbonaceous charring material was washed with double distilled water then washed with 0.1 N sodium bicarbonate solution for several times to remove the acid medium and soaked in 0.1 N sodium bicarbonate solution for 6 h to remove any acids in the carbonaceous charring. The product was once again washed with double distilled water and dried at $110\text{ }^{\circ}\text{C}$ in a hot air oven for 24 h and it was ground till to get fine powder [21-24]. The scanning electron microscope (SEM), micrographs of the sample was obtained using (Model: ZEISS, India). Fig. 1 shows that SGAC increases its adsorption capacity for methylene blue.

pH at Point of zero charge (pHPZC): The point of zero charge gives the available charges on adsorbent (SGAC), in this experiment electrolyte concentration kept constant in all the studies. 0.05 M KCl solution was the electrolyte, equal volume of electrolyte solution was taken in 250 mL of 12 no's of beakers, add constantly 20 mg of adsorbents in all the beakers containing KCl solutions and adjust initial pH values from 2-12. Stir the solution mixtures on a magnetic stirrer were allowed to equilibrate for 12 h at lab temperature. After completing the 12 h stirring, measure the final values of pH. Initial and final values of the pH were determined from the plots pH initial-pH final *v/s* pH initial [25].

Preparation of aqueous stock solution of methylene blue: The analytic grade methylene blue basic dye was chosen in the present work. To measure adsorptive properties of the prepared SGAC using methylene blue dye solution as model pollutant. The specification of the dye was in the following, C.I. of dye is 52015, chemical formula of $\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl}$, MW = 319.85 and $\lambda_{\text{max}} = 665$ nm is dark green powder [26]. The 100 mg accurately weighed methylene blue dye was dissolved in 1000 mL of double distilled water to prepare methylene blue aqueous stock solution (100 mg/L). The successive dilution of the stock solution was taken for the further experimental studies.

Batch adsorption experiment: The batch adsorption experiment was studied by the SGAC adsorbent with 50 mL of dye solution of 10 mg/L of concentration [27], was taken in a beaker, temperature at $25 \pm 3\text{ }^{\circ}\text{C}$ has a magnetic stirrer at constant rotation per minute. After agitation, dye solutions were

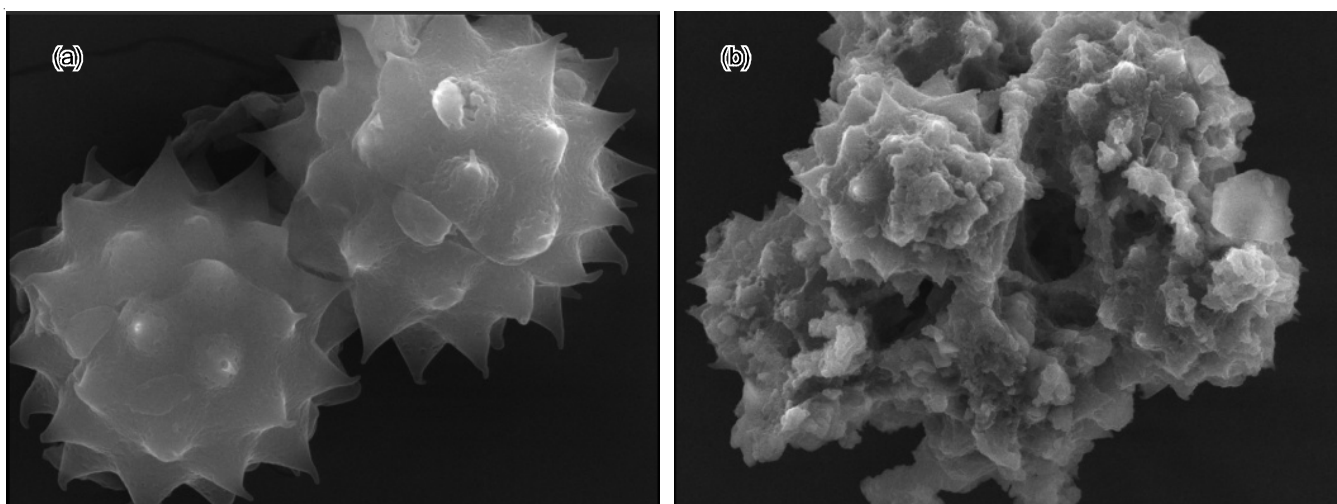


Fig. 1. SEM morphology of SGAC studied under SEM (a) before adsorption, (b) after adsorption

separated from the adsorbent by centrifugation. The supernatant liquid was taken and analyzed in spectrophotometrically by monitoring the absorbance before and after the treatment at wavelength of 665 nm [24]. The % adsorption rate of methylene blue can be calculated by the equation.

$$\text{Adsorption dye (\%)} = \frac{C_o - C_t}{C_o} \times 100 \quad (1)$$

where C_t and C_o are absorbance at various intervals of time (t) and original dye solution.

RESULTS AND DISCUSSION

Dosage effect: The present experimental observation in the effect of adsorbent dosage varied from 0.03-0.150 mg in the constant 50 mL of 10 ppm methylene blue dye solution concentration was taken in an 250 mL beaker at constant temperature $25 \pm 3^\circ\text{C}$, at neutral pH and stirring time of 30 min (Fig. 2). The maximum methylene blue adsorption taking place at 0.15 mg of SGAC per 50 mL methylene blue dye solution. The increase in the dye adsorption (99.6 %) with increase in SGAC dose, it shows maximum available adsorption sites filled by methylene blue molecules, if it increases further 0.18 mg it shows constant adsorption value [22].

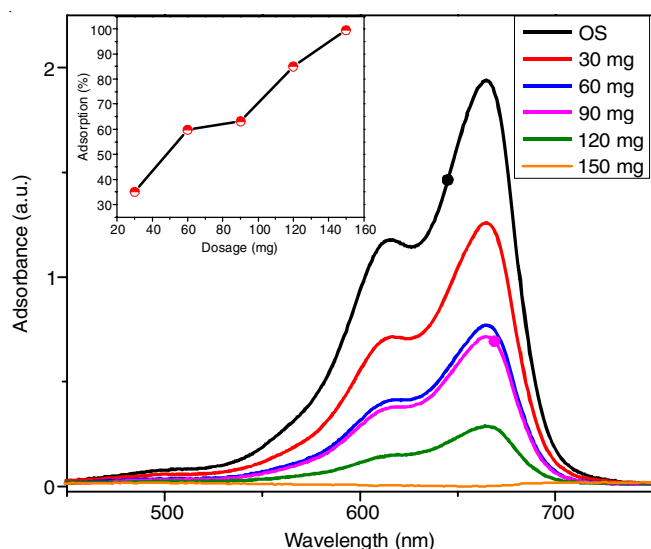


Fig. 2. UV-visible spectra for adsorption of methylene blue for different adsorbent dose, (a) 30 mg (b) 60 mg (c) 90 mg (d) 120 mg (e) 150 mg (f) O.S (original solution). (Inset) % adsorption of methylene blue vs. amount of SGAC

Effect of stirring time: The experimental results for adsorption property of SGAC on 10 ppm methylene blue solution at neutral pH and dosage of 0.150 g at different stirring time (3-18 min) are shown in Fig. 3. The stirring time can be attributed to the good rate of collision, the rate of collision between methylene blue and SGAC adsorbent at different intervals of time.

pH effect: pH effect is one of the major experimental parameters in the batch adsorption studies, it gives proper information about ionic charges of adsorbent (SGAC) and dye (methylene blue). In this study, pH of the solution varies from pH 2-12, at constant temperature ($25 \pm 3^\circ\text{C}$), at constant dosage

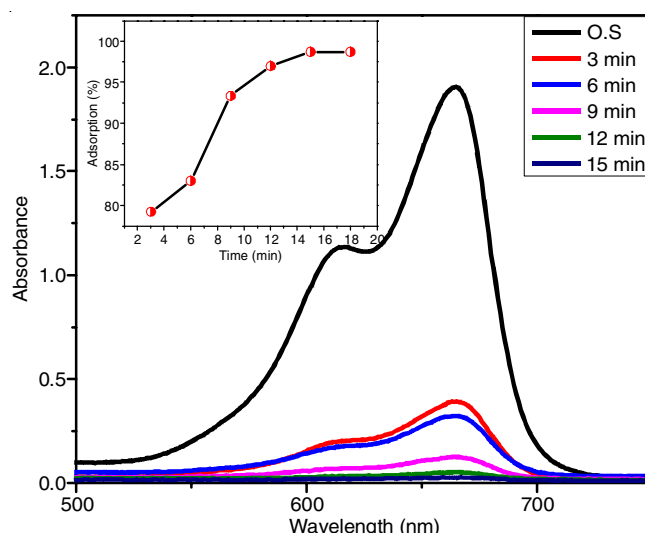


Fig. 3. UV visible spectra for adsorption of methylene blue for different contact time, (a) 3 min (b) 6 min (c) 9 min (d) 12 min (e) 15 min (f) 18 min and (g) O.S (original solution). (Inset) % adsorptions of methylene blue vs. stirring time

(0.150 mg) and at constant stirring time (18 min) as shown in Fig. 4. The maximum adsorption was observed at pH 4-6 (95 %) because the surface of SGAC adopts a slight positive surface charge (H_2SO_4 was used for activation), which contributes to enhanced uptake of basic dye molecules *via* electrostatic attraction, in accordance with an increase in the rate of adsorption. Further increases the pH 6-12, it was not remarkable adsorption takes place because adsorption of methylene blue at basic pH was accounted for the repulsion of negatively charged ions in solution containing methylene blue and SGAC.

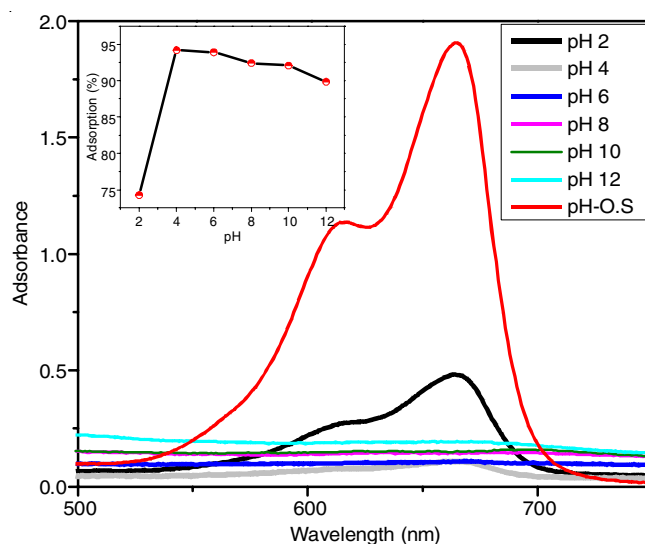


Fig. 4. UV visible spectra for degradation of methylene blue for different pH, (a) 2 (b) 4 (c) 6 (d) 8 (e) 10 (f) 12 (g) O.S (original solution). (Inset) % adsorption of methylene blue vs. pH variation

pH at point zero charge (pH pzc): The pHpzc experiments were performed with SGAC adsorbent. The pH range lower than the pHpzc value, exterior of SGAC is positively charged due to protonation, favouring the adsorption of anions. Other hand shows, SGAC exterior has a positive charge, which is special treatment to the adsorption of basic dye kind of

methylene blue [28]. In this observation pH_{pzc} of SGAC shows pH 6, which indicates the acidic nature of SGAC exterior. It was the evidence for the SGAC acid nature, because during the preparation of adsorbate we are using strong sulfuric acid, diagrammatical representation shown in Fig. 5.

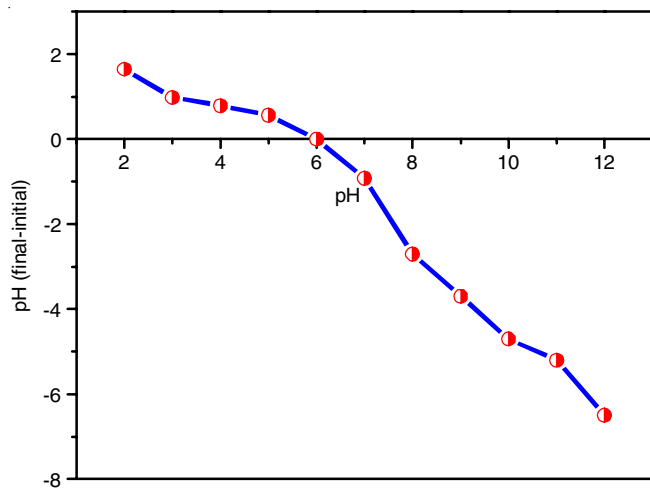


Fig. 5. Curve of point of zero charge

Effect of initial dye concentration: The lower dye concentration is suitable for maximum dye adsorption at constant stirring time at constant pH and temperature (25 ± 3 °C). Because dye concentration is low, strong monolayer adsorption taking place on exterior of adsorbent and all the binding sites are filled uniformly but in the case of more dye concentration, uneven adsorption takes place on the exterior surface of SGAC and also homo molecular adsorption takes place, resulting in the decrease in the adsorption efficiency (Fig. 6) [29,30].

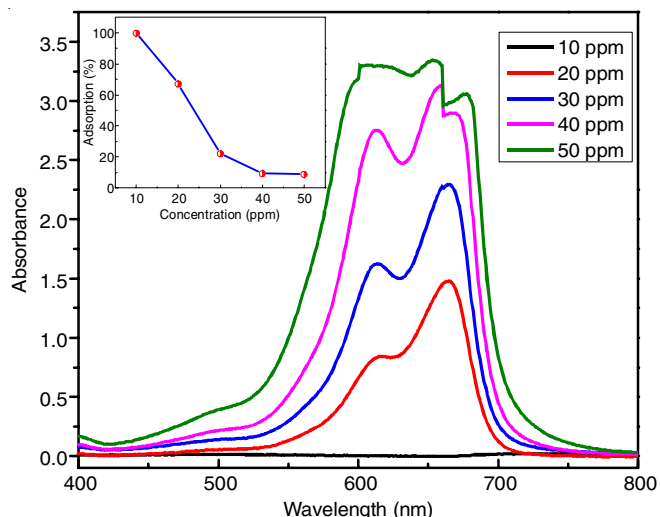


Fig. 6. Effect of dye concentration, (a) 10 ppm, (b) 20 ppm, (c) 30 ppm, (d) 40 ppm, (e) 50 ppm

Regeneration studies: All parameters of batch adsorption experiments completed, were collected some amount of used adsorbent. Washed with distilled water several times and dehydrated in hot air oven for 1 h at 80 °C and take 0.15 g of adsorbent (same as dosage studies), at a unchanging pH (neutral), concentration of 10 mg L⁻¹ (10 ppm) were centrifuged

and kept in UV-visible spectrometer, note down the regeneration absorption variations and calculate the % adsorption using eqn. 1 and this process was repeated for 4 to 5 times at the same condition. Fig. 7 shows percentage of adsorption decreases to 92.1 > 77.7 > 72.1 > 59.2 > 43.2 %.

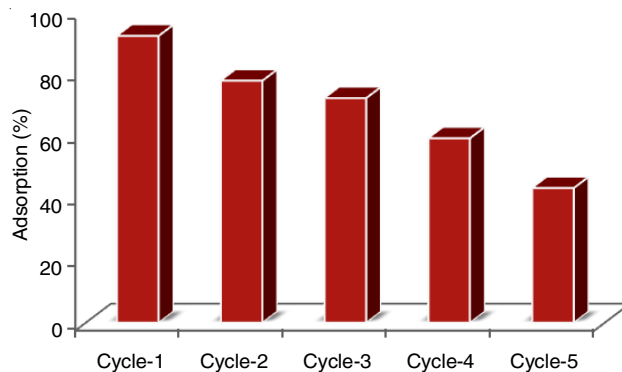


Fig. 7. Regeneration studies

Langmuir isotherm and Freundlich isotherm studies:

Adsorption isotherm equilibrium studies give the statistical quantity of maximum adsorption per gram of adsorbent [q_e (mg g⁻¹)] to the aqueous dye solution, the concentration [C_e (mg/L)] set time interval and at constant temperature [31]. This analysis has great evidence as of in cooperation of imaginary and realistic point of view to obtain well knowledge about adsorbent surface properties and adsorption mechanism. Langmuir and Freundlich adsorption isotherms are single layered adsorptions onto the exterior surface of SGAC with an integers of indistinguishable sites. The standards of b and Q_m were calculated using the slopes ($1/Q_m$) and intercepts ($1/bQ_m$) of the linear plots of C_e/q_e vs. C_e . The linearity of plots show that the adsorption followed Langmuir and Freundlich isotherm models. The Langmuir and Freundlich isotherm model sorption isotherm and its R^2 values are given in Table-1.

TABLE-1
ADSORPTION ISOTHERM VALUES OF
METHYLENE BLUE ONTO SGAC

Langmuir isotherm			Freundlich isotherm		
Q_m (mg/g)	R_L (L/mg)	R^2	K_F (mg/g)	$1/n$	R^2
61.72	1.542	0.966	0.375	0.079	0.9504

$$\text{Langmuir: } q_e = \frac{QbC_e}{1 + bC_e} \quad (2)$$

$$\text{Freundlich: } q_e = K_F C_e^{1/n} \quad (3)$$

The Langmuir isotherm (0.966) shows a high regression coefficient as compared to Freundlich isotherm (0.950), for methylene blue dye adsorption. Although the relatively higher R^2 values of the Langmuir adsorption point out that it is more preferable than Freundlich isotherm but the proximity of these values indicates that both of them are about to followed. It can be accomplished that most likely the exterior of SGAC contains heterogeneous moieties, which are homogeneously distributed on the exterior of SGAC, which accounts for both Langmuir and Freundlich adsorption isotherms [32].

TABLE-3
COMPARISON OF ACTIVATED CARBON BASED ON ITS ADSORPTION CAPACITY

Activated carbon treated by sulfuric acid	Q _{max} (mg/g)	Dosage (g)	pH	Ref.
Coconut leaves	127	0.15	6	[34]
Almond husk	37.2	0.50	5	[24]
<i>Parthenium hysterophorus</i>	39.7	0.40	7	[35]
Sunflower oil cake	16.4	0.20	6	[36]
Neem bark	5.71	0.02	2	[37]
Coconut tree sawdust	4.70	0.50	5	[38]
Bagasse	49.8-56.5	0.40	9	[39]
<i>Selenicereus grandiflorus</i> flowers activated carbon	16.17	0.15	4	Present paper

Adsorption kinetics studies: The effectiveness of technique requires an appreciative evidence getting from the kinetics studies. The kinetics study described the reaction pathways by the side of time to reach stability, kinetics is highly dependent on the substantial and substance uniqueness of the adsorbent like SGAC. The adsorption of dyes from (liquid) solutions phase to (solid) hard phase can be measured as a reversible reaction with stability established between the two phases [33].

The rate and mechanism of the adsorption process was measured using two special kinetic models, *i.e.* pseudo-first-order and pseudo second-order model. The pseudo-first-order model can be generally expressed by eqns. 7 and 8 as follows:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (7)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \quad (8)$$

The adsorption kinetic parameters of these two models are shown in Table-2. And also its linear regression coefficients, R². It shows that the R² values are greater (R² ≥ 0.837) for the pseudo-first-order kinetic model. Additionally, its q_e, cal values are in accord with the values of q_e, experimental. Therefore, pseudo-first-order kinetic model shows a better fit compared to the pseudo-second-order on exterior of SGAC adsorbent.

TABLE-2
KINETICS MODEL OF ADSORPTION OF
METHYLENE BLUE ONTO SGAC

Pseudo-first order kinetics		Pseudo-second order kinetics		
K ₁ (s ⁻¹)	R ²	Q _e (mg/g)	K ₂ (s ⁻¹)	R ²
0.0167	0.837	0.0654	-0.0659	0.8313

Comparison of activated carbon based on its adsorption capacity: From the literature survey, Table-3 listed some sulfuric acid treated activated carbon based on its adsorption capacity (Q_{max}), dosage (g/mL) and pH.

Conclusion

The present article study shows the usefulness of activated carbon from *Selenicereus grandiflorus* (SGAC) treated with conc. sulfuric acid as adsorbent for methylene blue dye from the aqueous solutions. According to literature survey, it is the fast adsorbent (18 min), maximum adsorption takes place at neutral pH (pH 4-6) and it requires 0.15 g adsorbent dosage. It minimizes the secondary pollutants because recycling process also done by using used SGAC. Langmuir and Freundlich isotherms studies give the evidence to the better monolayer adsorption and its regression coefficients are R² values 0.966 and 0.950. And also evaluate the pseudo first order and pseudo

second order kinetic models, it gives pseudo first order kinetics better fitted than the pseudo second order kinetics. Overall investigations of the present study, concludes that SGAC acts as an efficient adsorbent and its adsorption capacity Q is 16.17 mg of methylene blue dye per gram of SGAC in the batch experimental condition.

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