Original Research Article

Adsorption Studies of Methylene Blue on Gypsum

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ABSTRACT

To remove a cationic dye Methylene Blue from aqueous solution, Gypsum was investigated as an economical and efficient adsorbent. Batch kinetics and isotherm studies were carried out under varying experimental conditions of contact time, initial methylene blue concentration, adsorbent dosage and pH at room temperature. The nature of the possible Gypsum adsorbent and Methylene blue interactions was examined by the FTIR technique. The experimental data were analyzed by the Langmuir, Freundlich and Temkin models of adsorption. The adsorption isotherm data were fitted well to the Langmuir isotherm and the monolayer adsorption capacity was found to be 0.5339 mg/g at 25 ^{0}C. The data were also studied in terms of their kinetic behavior and was found to obey the first order equation.

Keywords: Adsorption, gypsum, adsorbent, methylene blue, dye, kinetics, isotherm.

1. INTRODUCTION

Dyes are widely used as coloring agents in a variety of industries, such as textiles, cosmetics, pulp mills, leather, dye synthesis, printing, foods, and plastics. Such dyeing industries consume large quantities of water and produces large volumes of wastewater from different steps in the dyeing and finishing process. [1] The wastewater discharged from a large number of industries, associated with dye stuff activities, contains high concentration of reactive dyes which causes a serious environmental problem. Some of these dyes have detrimental health effects. Effluents discharged from textile and dyeing industries are of low BOD and high COD. Disposal of this colored water into receiving water can be toxic to aquatic life. The dyes upset the biological activity in water bodies. These dyes also in water strongly absorb sunlight which decreases the intensity of light absorbed by water plants and phytoplankton, reducing photosynthesis and the oxygenation of water reservoirs. [2] The dye bearing effluents are considered to be a very complex and inconsistent mixture of many pollution substances ranging from organic-chlorine based pesticides to heavy metals and is considered to be recalcitrant, non-biodegradable and persistent. Industrial wastewater containing dyes is very difficult to treat, since the dyes are generally resistant to aerobic digestion. Various treatment methods including, physical, physico-chemical and chemical processes have been investigated for treating dye bearing effluents. All these methods have different color removal capabilities, capital costs and operating rates. [3] Adsorption is a well-known equilibrium separation process and an effective method for water

decontamination applications. Adsorption has been found to be superior to other techniques for water reuse in terms of initial cost, flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. [4-6] Activated carbon is the most widely used adsorbent to remove dyes in wastewater with great success. However, its widespread use is restricted due to its high cost. [7] The need to achieve high quality treatment at low cost and with high efficiency has led researchers to search for new sources inexpensive, readily available adsorbent materials.

In the present study, Gypsum was chosen as it is found abundantly in nature, is cheap as compared to other adsorbents such as activated coal or inorganic substances used for adsorption. Gypsum as an alternative low-cost adsorbent for the removal of Methylene Blue from aqueous solutions due to the reason that many textile manufacturers use dyes that release aromatic amines (e.g. benzidine, toluidine) and are potential carcinogens. [8-18] In this study, the effect of different parameters such as pH, contact time and initial dye concentration were investigated. Finally, the isotherm and kinetics as well as the diffusion parameters for the adsorption of Methylene blue dye onto the gypsum are evaluated. [19-32]

2. Experimental: In this experiment Methylene Blue dye (Waldeck–Gmbh & Co.), Gypsum, NaOH (BDH England), HCl (BDH England), Distilled H_2O are used as required materials. Electric Balance (Sartorrius Basic), UV–Visible recording Spectrophotometer (UV- 1800, Shimadzu, Great Britain), Magnetic Stirrers (MM 2A), Centrifuge Machine (IEC SPINETTE Centrifuse, 300sec.), pH meter (Model pH, S-25, REX), All types of beaker, volumetric flask, conical flask, measuring cylinder, reagents bottles, pipette, burette, were made of Pyrex glass England are used as essential apparatus.

2.1. Physical characteristics and molecular structure of Methylene Blue (MB)

Molecular formula: $C_{16}H_{18}N_3SCl$, Molar mass: 319.85 g/mol Melting point : 100-110 °C (with decomposition), Boiling point: Decomposes, λ_{max}: 664 nm, The molar extinction coefficient: 77118 L mol^{-1} cm^{-1}, IUPAC name: 3,7-bis (Dimethylamino)-phenothiazin-5-ium chloride.
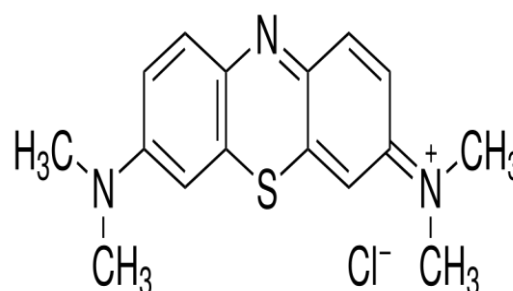

Fig.1: Structural formula of Methylene blue

2.2. Physical characteristics of gypsum:

Chemical occurrence: $CaSO_4 \cdot 2H_2O$, Gypsum content: 97%, Bulk density: 0.87 g/cm^3, Particle density: 2.5 g/cm^3, Moisture content: 3%, BET surface area: 5.67m^2/g, Average pore diameter: 10.79Å, Micropore volume: 3.59×10^{-3} cm^3/g, Mesh size: <75µm. [33]

2.3. Experimental techniques for measurement of MB adsorption on adsorbent:

The adsorption of MB on Gypsum adsorbent was monitored spectrophotometrically by taking the absorbance of MB from its aqueous solution at $\lambda = 664$ nm, before and after the addition of adsorbent. MB dye solutions of chosen concentrations were made in deionized water. Preliminary experiments were carried out on this dye solution to determine the working concentration range in the Lambert–Beer region. The MB dye solution is highly colored and shows an intense absorption peak in the visible region. For this reason, an adsorption process can be targeted to characterize the removal of MB dye from the solution and commercially available gypsum powder

was obtained from the local market. Firstly, MB dyes stock solution of 1×10^{-3} M was prepared in 250 mL of deionized water in a flask followed by necessary dilutions of this stock solution. For each experimental run, 25 mL of MB solution of known concentration, known pH and a known amount of the adsorbent were taken in a reagent bottles. Then the solutions within the reagent bottle were shaken for a certain time by magnetic stirrer at a constant speed at room temperature.

The bottles were allowed to settle down and aliquot portion of solution from each bottle were taken in test tubes and centrifuged. The centrifugates were collected carefully and then monitored instantaneously on a spectrophotometer for absorbtion. The change in absorbance of the MB dye solution was then used to calculate the dye concentration in solution and the percentage adsorption of the dye on gypsum.

$$Dye(MB)adsorption(\%) = \frac{C_o - C_t}{C_o} \times 100 \qquad (1)$$

Where, C_0 and C_t (both in mol/L) are the liquid-phase concentrations of initial dye and at any time t respectively.

Equilibrium adsorption capacity was calculated from the relationship

$$q_e = \frac{(C_o - C_e)V}{W} \times 100 \qquad (2)$$

Fourier Transform Infrared (FTIR) analysis was applied on gypsum and MB dye adsorbed gypsum to determine the surface functional groups, where the spectra were recorded from 4000 to 400 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1. FTIR Characterization: Fig-2 represents the FTIR spectra of gypsum before dye adsorption and after dye adsorption. The spectra display a number of absorptions peaks. In **Fig-2(a)**, the peaks between (1050-1073) cm^{-1} indicate the S=O bonds and (2245-2500) cm^{-1} is due to the presence of S-H bonds. The peaks around 1375 and 1185 cm^{-1} indicates the S=O symmetric and asymmetric stretch respectively. The existence of O-H band occurs in the range between 3200-3400 cm^{-1} and bands at 650 cm^{-1} indicates the S-O stretching. However, the highly intense bands at (1350-1105) cm^{-1} and (1375-1300) cm^{-1} may be the overlapping of C-N (amine) and sulfonamide groups after dye adsorption which is shown in **Fig-2(b)**. The band at 785-580 cm^{-1} is due to the C-Cl bond sine MB contain Cl atom. Thus, it is inferred from IR spectrum that adsorbent contains SO groups that are responsible for the electrostatic attraction of MB cations. Therefore, the functional groups on the gypsum surface can attract ionic dye molecules of opposite charge or lone pair of electrons which lead to increase in dye removal efficiency.
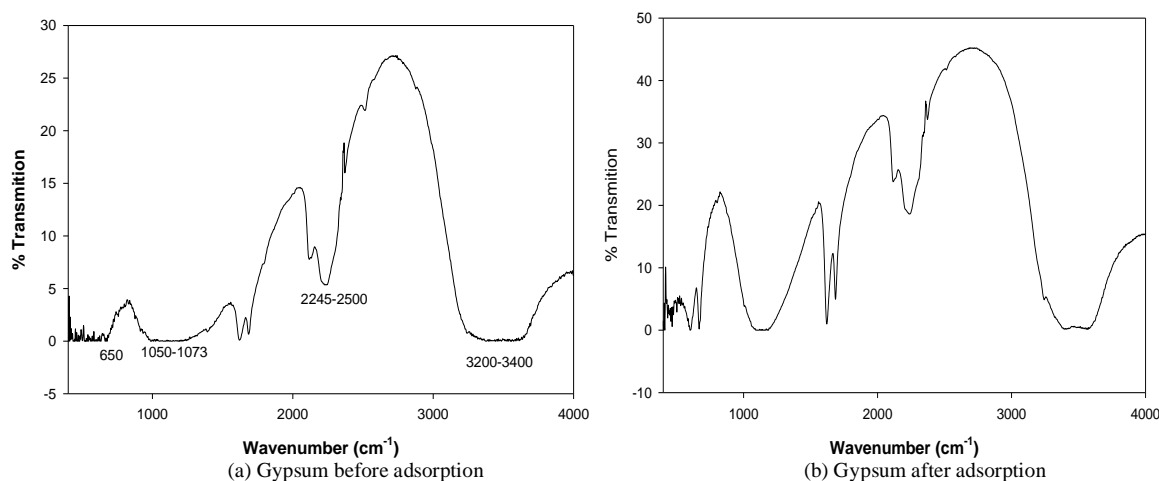

(a) Gypsum before adsorption (b) Gypsum after adsorption
Fig.2. FTIR spectra of Gypsum (a) before MB dye adsorption and (b) after MB dye adsorption

3.2. Influencing parameters of MB adsorption:

The functional group of MB dye was influenced by different parameters by adsorption onto Gypsum. The affected parameters on MB were shown (such as pH, concentration, absorbent dosage etc.).

3.2.1. Effect of Ph:

The pH value of the solution is an important controlling parameter in the adsorption process, and the effect of pH on percentage adsorption of Methylene Blue on gypsum is shown **Fig.3**. The adsorption of MB dye was found to decrease at low pH values. This may be due to the fact that at low pH values, the H^+ ions may compete with dye molecules for the adsorption sites of the adsorbent, thereby inhibiting the adsorption of dye.
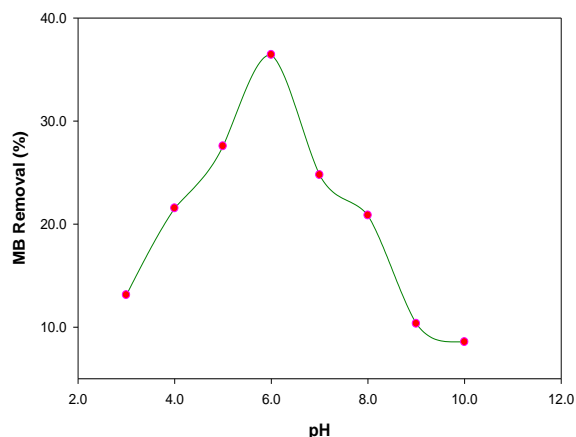

Fig.3. Effect of pH on MB adsorption on gypsum [MB] =2.0×10^{-5} M, Gypsum=0.025 g, contact time 60 min, at room temperature.

The adsorption percentage is maximum at the natural pH value of the dye solution (pH 6.0). On increasing the pH values, the adsorption process slows down. This could be due to the reason that at higher pH levels, excess OH^- ions compete with dye molecules for adsorption sites on the adsorbent surface resulting in less adsorption. [34]

3.2.2. Effect of adsorbent dosage on MB adsorption:

The effect of gypsum dosage on the removal of MB was studied for an initial dye concentration of 2.0×10^{-5} M, by varying the dosage from 0.015 to 0.35 g, keeping all other parameters constant. **Fig.4** shows the effect of gypsum dose on

the % removal of MB. It was observed that the % removal increased with increase in adsorbent dose.
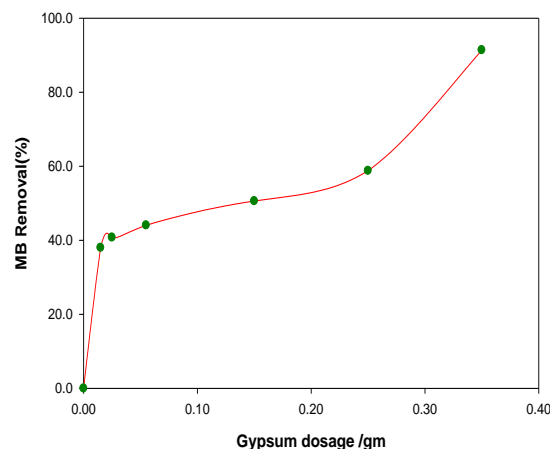
Fig.4. Effect of adsorbent dosage on the adsorption of MB on gypsum, [MB]=2.0×10-5 M, pH 6.0, contact time 60 min, at room temperature.

At equilibrium time, the % removal was increased from 38.00% to 91.40 % for an increase in gypsum dose from 0.015 to 0.35 g. The increase in % color removal was due to the increase of the available sorption surface and availability of more adsorption sites.

3.2.3. Effect of contact time:

The effect of contact time for the removal of MB dye by gypsum adsorbent is shown in **Fig. 5**.
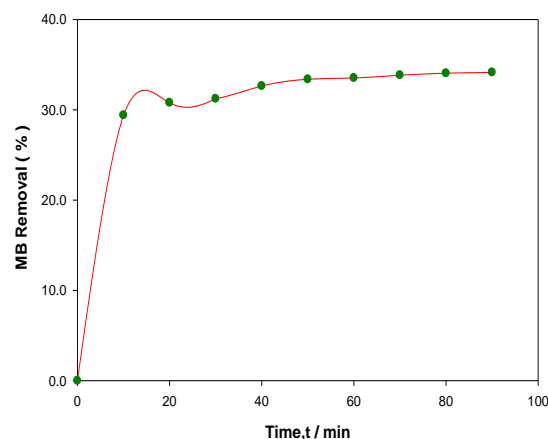
Fig.5. Change in adsorption of MB on Gypsumwith time, [MB] =2.0×10^{-5} M, Gypsum=0.025 gm, pH 6.0, contact time 90 min, temperature=25^{0}C.

The amount of MB dye adsorbed on gypsum increases with time and later becomes constant. The rapid adsorption was observed during the first 10–30 min is probably due to the abundant availability of active sites on the gypsum surface with

a gradual decrease of these sites with time and therefore adsorption becomes less with time and finally becomes constant. [35]

3.2.4. Effect of initial MB dye concentration:

Changing the concentration of MB dye solution caused a change in adsorption of dye. The parameter, initial concentration, provides an important driving force to overcome resistances encountered when all molecules were transferred between the aqueous and solid phases. Effect of initial MB dye concentration on adsorption was investigated at concentration ranging from 1.0 to 10.0mol /L as shown in **Fig.6**.
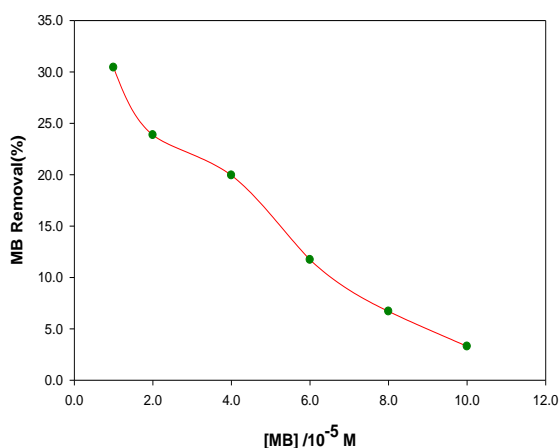

Fig.6. Change in adsorption of Methylene Blue on gypsum with its concentration, amount of gypsum = 0.025 g, contact time = 60 min, pH 6.0, temperature =25^{0}C.

By increasing the initial MB dye concentration the percentage of dye removal was decreased. This is because that at low concentrations, the adsorption sites can be occupied by dye molecules, but when concentration of dye was increased in solution, the active sites on the adsorbent surface are already occupied, thereby causing less removal of dye from solution. Also the steric repulsion between the dye molecules can slow down the adsorption process. [19]

3.3. Adsorption isotherms study:

Adsorption isotherm is basically important to describe how solutes interact with adsorbents and is critical in optimizing the use of adsorbents. Adsorption isotherms can be used to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface at equilibrium.

In order to discover the adsorption capacity of gypsum, the experimental data points were fitted to the Langmuir, Freundlich, and Temkin, isotherm equations and the constant parameters of the isotherm equations were calculated. The main difference between these three isotherm models is in the variation of heat of adsorption with the surface coverage. Langmuir model assumes uniformity, Freundlich model assumes logarithmic decrease and Tempkin model assumes linear decrease in heat of adsorption with surface coverage. [20]

The Langmuir equation, which is valid for monolayer adsorption onto a completely homogenous surface with a finite number of identical sites and with negligible interaction between adsorbed molecules, is represented in the linear form as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \qquad (3)$$

Where K_L is the Langmuir adsorption constant and Q_m is the theoretical maximum adsorption capacity. **Fig. 7** shows the Langmuir (C_e/q_e vs. C_e) plots for adsorption of methylene blue at room temperatures.
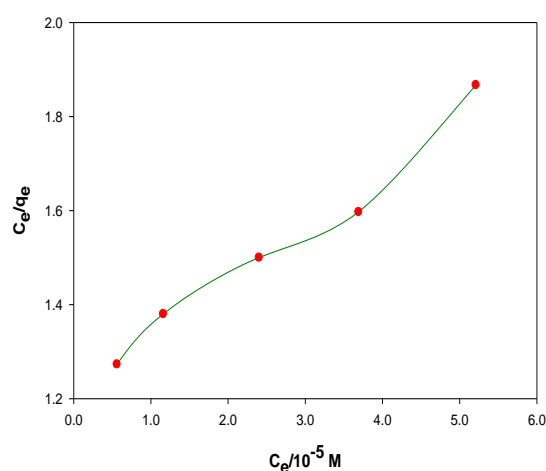

Fig.7. Langmuir isotherm of the adsorption of MB on Gypsum, Gypsum=0.025g, contact time 60 min, pH=6.0, temperature =25^{0}C.

Experimental data for the MB dye adsorption on gypsum was also subjected

to Freundlich equation, which has the following form

$$ln\,q_e = ln\,k_f + \frac{1}{n}\,ln\,c_e \qquad (4)$$

Where, q_e is the concentration of MB dye adsorbed on gypsum and c_e is the bulk concentration of dye; K_f and n are characteristic constants for a sorption system.

Fig. 8 shows the Freundlich (Ln q_e vs. LnC$_e$) plots for adsorption of Methylene Blue at room temperatures.
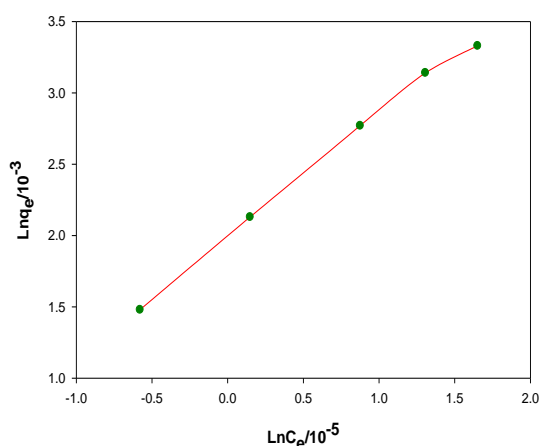
Fig.8. A plot of Lnq$_e$ vs. LnC$_e$ for adsorption ofMB on Gypsum at room temperature, Gypsum = 0.025 gm, [MB] = 2.0×10^{-5} M, contact time 60 min, pH 6.0,
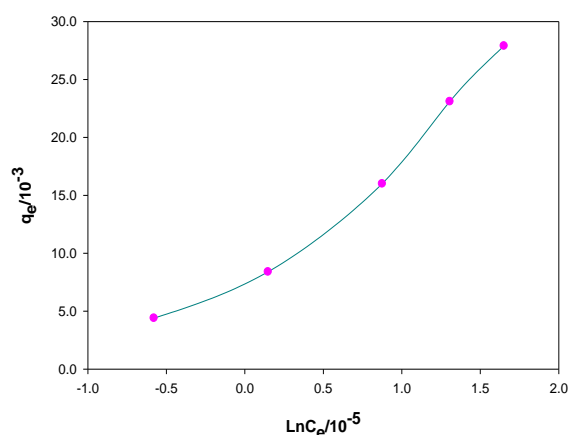
The adsorption data was also analyzed by using the Tempkin equation, which is mathematically given by:

$$q_e = RT\,ln\,A\,\left(\frac{C_e}{b}\right) \qquad (5)$$

In its linear form the above equation can be written as:

$$\boldsymbol{q_e} = k\,ln\,A + k\,ln\,C_e \qquad (6)$$

Where k is Tempkin isotherm energy constant (RT/b) and A is the Tempkin isotherm constant. A plot **of q$_e$** versus LnC$_e$ at studied concentration as shown in Fig. 9.

Fig. 9: Tempkin isotherm of MBadsorption On Gypsum, Gypsum= 0.025 gm, [MB] = 2.0×10-5 M, contact time 60 min, pH 6.0,

Table 1: Data for the adsorption of initial MB dye concentration on the gypsum adsorbent for adsorption equilibrium study at pH 6.0, contact time 60 min, Gypsum = 0.025 gm, temperature 25^{0}C.

Initial Conc. $\times 10^{-5} M$	Initial Absorbance	Final Absorbance	Eqm. Conc(C_e) $\times 10^{-5} M$	$q_e \times 10^{-3}$ mol g^{-1}	$q_e/C_e \times 10^{-3}$ g L^{-1}	LnC$_e$/ 10^{-5}	Lnq$_e$ /10^{-3}
1	0.621	0.432	0.560	4.400	1.273	-0.579	1.48
2	1.174	0.894	1.160	8.400	1.380	0.148	2.13
4	2.311	1.850	2.400	16.00	1.500	0.875	2.77
6	3.223	2.845	3.690	23.10	1.597	1.306	3.14
8	4.309	4.021	5.210	27.90	1.867	1.651	3.33

The calculated value of Langmuir adsorption constants K$_L$, the theoretical maximum adsorption capacity Q$_m$,

Freundlich constants K$_f$ (adsorption capacity) and n (adsorption intensity) and the Temkin constants K are given in table 2.

Free energy of adsorption was found as, $\Delta G^0 = -RTLnK_L = -6.19$ KJ. The negative value of ΔG^0 indicates the spontaneous adsorption on the surface. The isotherms were almost linear over the whole concentration range and correlation

coefficients were good (R$^2 > 0.96$).The value of adsorption intensity (n) is 0.470.

Table 2: Langmuir, Freundlich and Temkin isotherm model constants and correlation coefficients for adsorption of Methylene Blue onto Gypsum.

Isotherm parameters	Parameters
Langmuir	
K$_L$ (Lg^{-1})	12.14
Q$_m$ (mg/g)	0.5339
R	0.98648
Freundlich	
K$_f$ (Lg^{-1})	84.55
n	0.47
R	0.99683
Temkin	
K (Lg^{-1})	10.69
R	0.96365

3.4. Adsorption kinetics

The adsorption kinetics of the MB dye adsorption on gypsum was also analyzed by using kinetic model. The adsorption rate is strongly influenced by several parameters related to the state of the solid, generally having very heterogeneous reactive surface, and to the physico-chemical conditions under which adsorption was carried out.

In order to investigate the adsorption processes of Methylene Blue on the adsorbents, the first-order kinetic models were studied,
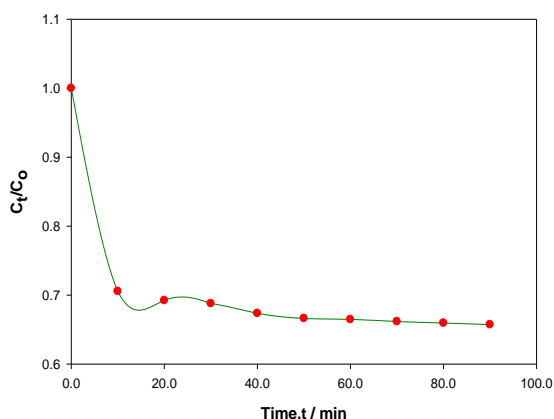
$$C_t = C_o \ e^{-kt} \qquad (7)$$

In the linear form above this equation can be written as:

$$-ln \frac{C_t}{C_o} = kt \qquad (8)$$

Where, C_o and C_t are amounts of dye adsorbed at equilibrium and at any time t, k is the first order rate constant and t is the time.

The concentration of MB adsorption on Gypsum was decreased with the contact time as shown as in **Fig.10** from **Fig.11** the value of rate constant k was found to be 8.66×10^{-4} min^{-1} and the adsorption data was fitted linearly to first order kinetics over the entire region, when the pH of the MB solution is 6.0.
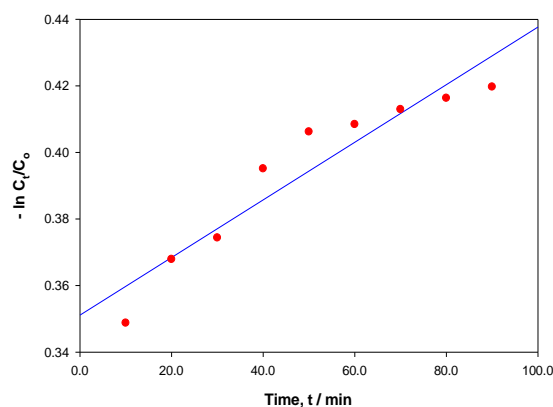

Fig.10. A plot of C_t/C_o vs. Time (t) for adsorption of MB on Gypsum.

Fig.11. A plot of $ln[C_t/C_o]$ vs. Time(t) for the First order reaction at room temperature

4. CONCLUSION

The adsorption of MB from aqueous solution using Gypsum as low-cost adsorbent was investigated under different experimental conditions in a batch process. Methylene Blue was removed from aqueous solution onto gypsum surface at room temperature. The equilibrium data were analyzed using the Langmuir, Freundlich and Tempkin isotherm models. The Langmuir adsorption isotherm was found to have the best fit to the experimental data with maximum adsorption capacity of 0.5339 mg/g. The kinetics of MB adsorption onto Gypsum was followed the first-order kinetic equations.

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