



Cassia Alata Seed Activated Carbon for the Removal of Synthetic Dye Methyl Orange from Waste Water

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ABSTRACT

In this study, the activated carbon was prepared from Cassia alata seeds using Hydrochloric acid. Removal of Methyl Orange (MO) was assessed by varying parameters like pH, adsorbent dose, concentration, contact time and temperature. The adsorption efficiency of the prepared activated carbon was estimated by using various isotherms and kinetic equations. The isotherm parameters of the process were determined by using Langmuir and Freundlich isotherm equations. The kinetic parameters were predicted with pseudo first order and pseudo second order equations. The kinetic data confirmed to the pseudo second order kinetic model, suggesting that the rate limiting steps may be chemisorptions. The adsorbent material characterization was done by Fourier Transform Infrared Spectroscopy (FTIR) and morphology of the surface adsorbent was identified with Scanning Electron Microscope (SEM).

Keyword: Activated carbon, Cassia alata seed, Methyl Orange, Isotherms

INTRODUCTION

Carbon is extremely widespread in nature. It is an essential constituent of all living organisms. Activated carbon can be considered as a black solid substance resembling powdered charcoal [1]. Activated carbon differs from carbon by its surface. Activated carbon's surface area has millions of porous structure [2]. It is used as an adsorbents in many industries for the

removal of dye [4]. Activated carbon is obtained from the sources with high carbon content e.g. Plant seeds, various bark powder and stem of plants. It is also produced from variety of natural and synthetic substances. In general activated carbons are prepared by physical and chemical activation [4]. Preparation of activated carbon is very easy and it is low cost [5]. If it is prepared from low cost non-conventional sources it should be economical [6].

Dye stuffs from industrial effluent is highly toxic because dyes are carcinogenic in nature, when it is mixed with the water sources it affects aquatic plants and animals. Methyl orange is one of toxic substance present in waste bodies. Methyl orange is an anionic azo dye [7]. The azo dyes possess nitrogen in molecule. The reactivity is due to presence of functional group, ie. azo group (N=N) It is soluble in water [8]. Methyl orange is a synthetic dye. It is used as a colouring agents in textile industry, food printing, paper manufacturing, pharmaceutical and research laboratories [9]. Methyl orange causes increased heart rate, vomiting, cyanosis, Jaundice and Skin allergies for human beings. Removal of this dye pollutant is very much important to reduce harmful effects. Many methods have been used to remove the dye. e.g., Adsorption, precipitation, electrical remediation, oxidation and biological degradation [10]. All the above techniques adsorption is a efficient, simple and flexible method [11]. Because adsorption method will not produce any harmful by products.

A detailed literature survey shows that Activated Carbon of Cassia alata seed carbon is not used as an adsorbent material to remove Methyl Orange dye. Our attention was focussed on Cassia alata plant seeds [12] it is also known as Senna alata belongs to the family Leguminosae [13]. It is an agricultural waste material. This seeds are numerous and flat and has high carbon content. In the present work activated carbon is prepared from the seeds of Cassia alata and utilised for the removal of Methyl Orange azo dye by the process adsorption. From the adsorption measurements the first and second order kinetic equations are evaluated. The functional group of the adsorbent was analysed using Fourier Transform Infrared Spectroscopy (FTIR) [14].

MATERIALS AND METHODS

Materials

Cassia alata seeds were collected from Thiruvannamalai District, Tamil Nadu. The chemical substances including Hydrochloric acid and Sodium Hydroxide, Acetone, Methyl Orange were purchased from Sigma Aldrich (Chennai). The pH of solutions was adjusted with dilute Hydrochloric acid and sodium hydroxide solutions using a digital pH meter.

METHODS

Preparation of adsorbent

After separation of seeds from pods of cassia alata plant the remaining waste was carefully washed with deionised water to remove impurities then dried and carbonized in a furnace at 450°C for 5 hrs. The carbonized sample was transferred into 500ml sulphuric acid solution (0.1 N) for 42 hrs. The suspension was filtered and the modified samples were washed several times with distilled water until the pH of the washings become neutral. Finally, the modified activated carbon was dried in an electrical oven at 105°C for 12 hrs. The modified activated carbon-based Cassia alata seeds was crushed and then sieved to have a uniform particle size for the adsorption experiments.

Adsorbate

The stock solutions of methyl orange (0.1g/L) were prepared in distilled water and the working concentrations were also obtained with dilution of the stock solutions. The molecular formula of methyl orange (p-dimethylamino-azobenzene sulfonic acid) is, C₁₄H₁₄N₃NaO₃S and Formula weight is 327.34. The structure of methyl orange is shown in Fig. 1. The

suspensions containing adsorbent and adsorbate were mixed using an orbital shaker at 200rpm.

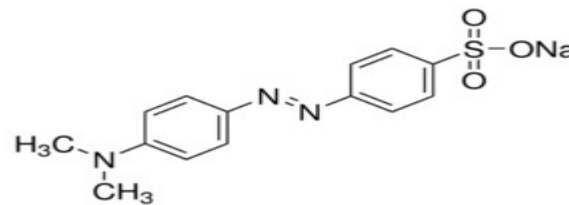


Fig.1 Structure of Methyl Orange

Experimental Methods

In each adsorption experiment, 60 ml of dye solution with a known concentration was added to 0.3g of adsorbent in a 250 ml glass-stoppered bottle at 30 ± 0.5°C, and the mixture was stirred on a mechanical shaker operated at 150 rpm for 60 min. Kinetics of adsorption was determined by analyzing adsorptive uptake of the colour at different time intervals. The samples were withdrawn during stirring at preset time intervals, and the adsorbent was separated from the solution by centrifugation (Research Centrifuge, Remi Scientific Works, Mumbai) at 4000 rpm/min for 5 min. The absorbance of the supernatant solution was estimated to determine the residual dye concentration, and was measured before and after treatment with double beam spectrophotometer (λ_{max} 450nm) (HITACHI U 2000 Spectrophotometer). Isothermal studies were conducted with different amount of adsorbate (20mg/l to 100mg/l). All experiments were carried out twice, and the concentrations given are average values. The effect of pH was observed by studying the adsorption of dye over the pH range from 3 to 8. The pH of the dye solution was adjusted by using NaOH or HCl solution and a pH meter. The sorption studies were carried out at different temperatures (30⁰, 60⁰, 90⁰,120⁰C). This is used to determine the effect of temperature. The amount of sorption at time t, q_t (mg/g), was calculated using the following formula:

$$q_t = \frac{(C_0 - C_t)V}{w} \quad (1)$$

where C_t (mgL⁻¹) is the liquid phase concentrations of dye at any time, C₀ (mgL⁻¹) is the initial concentration of the dye in solution.. V is the volume of the solution (L) and W is the mass of dry adsorbent (g).

The amount of equilibrium adsorption, q_e (mg /g), was calculated using the formula

$$q_e = \frac{(C_0 - C_e)V}{w} \quad (2)$$

where C_0 and C_e (mgL^{-1}) are the liquid-phase concentrations of dye initially and at equilibrium.

The dye removal percentage can be calculated as follows:

$$\% \text{ of dye removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (3)$$

where C_0 and C_e (mgL^{-1}) are the initial and equilibrium concentrations of the dye in solution.

RESULTS AND DISCUSSION

FTIR spectrum

The FTIR spectra of activated carbon after the adsorption of MO is illustrated in Fig.2. According to this spectra the broad and intense absorption peak at 3428cm^{-1} corresponds to the O-H stretching vibrations of MO. The weak peak around 2925 cm^{-1} caused by C-H vibration. The weak peaks around 1600 cm^{-1} are due to the C-C stretching. Bands in the range of 1386cm^{-1} are attributed to COO^- symmetric stretching vibration. The another band at 1117cm^{-1} is attributed to C-O stretching of ether group and 1035cm^{-1} is due to C-O stretching of carboxylic acid group [15].

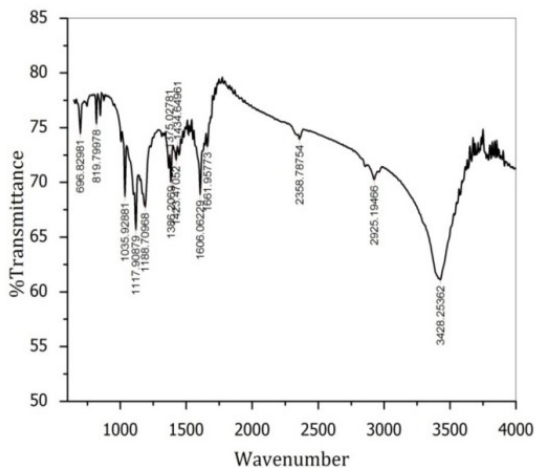


Fig.2 Infrared Spectrum of Methyl Orange loaded activated carbon.

SEM

The SEM image of the prepared activated carbon is shown in Fig.3, many large pores were clearly found on the surface of the activated carbon. The well developed pores had led to the large surface area and porous

structure of the activated carbon. Well-developed cavernous porous structures were clearly visible in micrographs which may assist in accommodating large amount of dye molecules from synthetic aqueous solutions to the adsorbent surface.

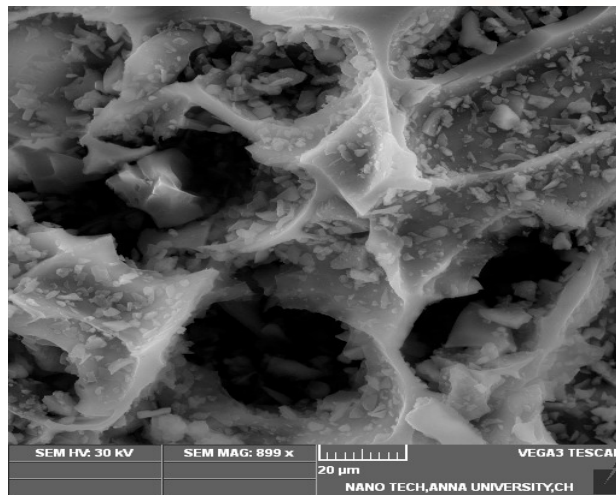


Fig. 3 SEM image of Cassia alata seed Activated carbon

Influence of system pH on adsorption

It is well known that the pH is one of the parameter plays an important role that affects the surface charge of adsorbent, the degree of ionization of dyes the structure of the dye molecule, the dissociation of functional groups on the active sites of the adsorbent, and the extent of rate of adsorption. The initial pH values of the solution has more influence than the final pH value [16]. The percentage of Methyl Orange adsorption was studied as a function of pH in the range of 3-8. The minimum adsorption was 88% at pH 3.0 and maximum adsorption was 90.2% at pH 5.0 for 60mg of initial concentration of dye solution. This increase in percentage of removal is due to strong electrostatic force of attraction between the adsorbate and adsorbent. This is shown in the Fig. 4.

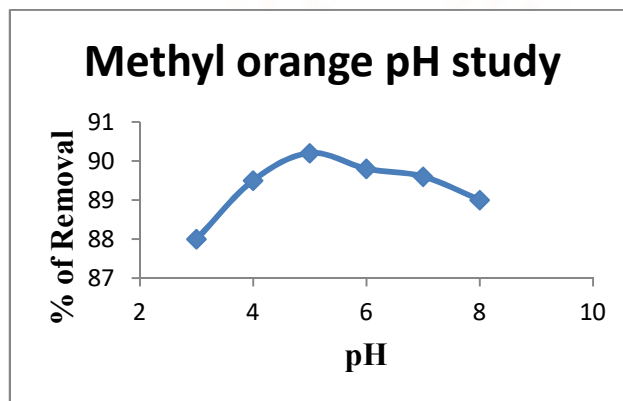


Fig.4 Effect of pH on MO dye sorption.

Effect of dye concentration

The effects of initial dye concentration for the adsorptive removal of Methyl Orange dye from aqueous solution over activated carbon were studied and the results are given in figure 5. The concentration of dye solution varied from 20mg/L to 100mg/L. It is observed that the maximum percentage of removal occurs when the initial concentration of dye is 20mg/L. When the concentration of dye is increased gradually the percentage of dye removal also decreases gradually. This is due to poor interaction between the dye and activated carbon. While the maximum removal of Methyl Orange dye is 94.2% for 20mg/L of initial concentration. In the present study 86.81% of dye removed when the initial concentration of dye is 60mg/L. Similar conclusions for the removal of Methyl Orange from aqueous solution using activated carbon prepared from Prosopis Juliflora reported [7].

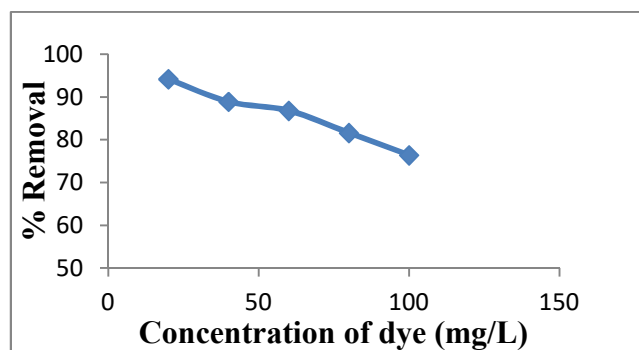


Fig.5 Effect of Dye Concentration on MO dye sorption

Effect of adsorbent dose

Fig. 6, shows the effect of adsorption of Methyl Orange dye as a function of an increase in the dosage of activated carbon prepared from cassia alata. The adsorbent dose is also an important parameter because it determines the capacity of a adsorbent for a given initial concentration. Studies were conducted by varying the amount of the adsorbent dosage ranging from 0.1g to 0.7g with a fixed concentration of Methyl Orange dye 60mg/L. When the adsorbent dosage increased from 0.1g to 0.3g the percentage of removal increased from 87% to 91.5%. This is due to the increase of surface area and increase of number active sites. The percentage of removal gradually decreased when the adsorbent dose is varied from 0.4g to 0.7g. This is due to aggregation of adsorbent takes place due to increase in the concentration of adsorbent. The experimental result showed that 0.3g of adsorbent dosage was the optimum quantity for the removal of

methyl orange. Therefore 0.3g was optimum dosage chosen for all further studies.

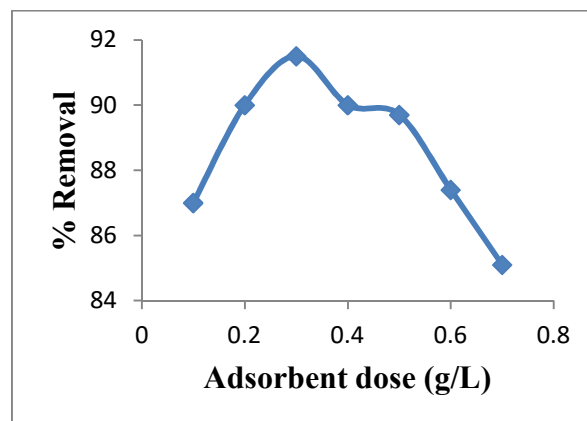


Fig.6 Effect of adsorbent dose on MO dye sorption.

Effect of Temperature

The dependence of temperature on the % of removal of dyes is investigated in different temperature ranges from 20°C to 40°C with the increment 5°C shown in Fig.7. It was found that at 30°C the adsorption is maximum (pH=5). This result shows that the maximum percentage of removal takes place at 30°C. When the temperature is raised from 30°C to 40°C the percentage of removal is decreased slightly. From this observation we can conclude that the increase of temperature weaken the physical forces of attraction between the active sites on the adsorbent and the dye.

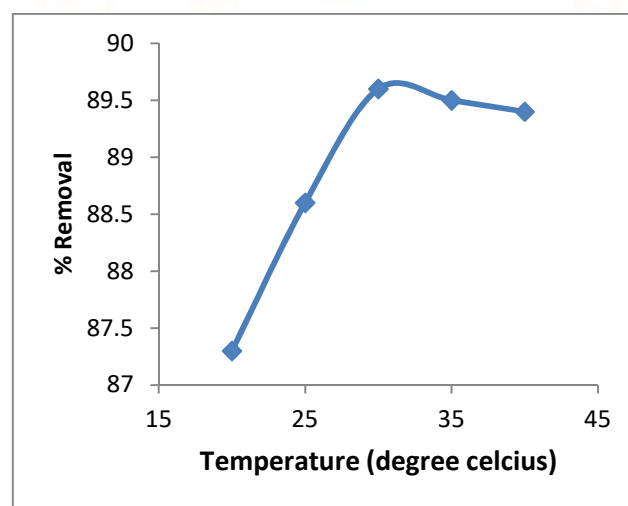


Fig.7 Effect of Temperature on MO dye sorption.

Effect of contact time

The decolourization of dye was studied as a function of agitation time at room temperature. The decolourization is using various time intervals as 20 min. to 80 min. with the increment range of 20 minutes. From the result it is observed that the percentage of removal increases

with the increase in contact time. About 20 minutes itself the percentage of dye removed is 86.8%. About 40 minutes the percentage of dye removed is 89.9%. When the contact time increased from 40 to 80 minutes the percentage removal not varied so much. No appreciate increase in percentage of removal indicates that all the active sites are occupied by the dye particles. This is shown in Fig.8.

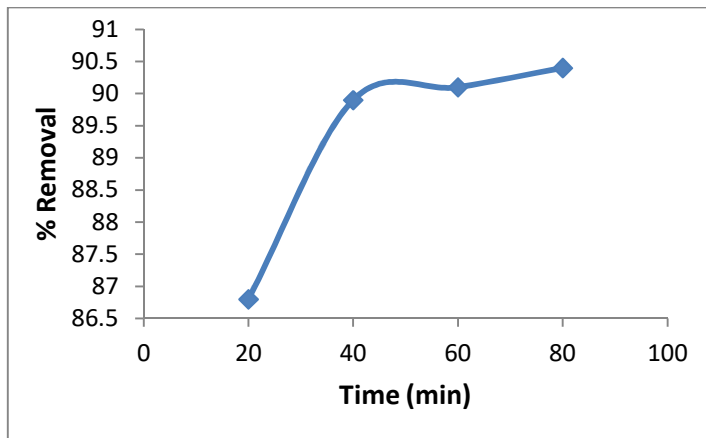


Fig.8 Effect of Contact time on MO dye sorption.

Adsorption studies

The equilibrium adsorption isotherm of methyl Orange from an aqueous solution using activated carbon was investigated in batch. In this study the Freundlich and Langmuir isotherm, were applied to understand the dye-activated carbon interaction. The Langmuir isotherm is based on the assumption of monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform energies of adsorption. The Freundlich isotherm describes a heterogeneous system and reversible adsorption and is not restricted to monolayer formation [17].

Freundlich Isotherm

The logarithmic form of Freundlich isotherms equation is

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Where q_e is the amount of dye adsorbed per unit mass of the adsorbent (mg/g), C_e is the equilibrium liquid-phase concentration of the dye (mg/L), the K_F represent quantity of dye adsorbed in mg/g for unit concentration of dye and $1/n$ is measure of adsorption density. Values of $n > 1$ represent the favourable adsorption condition. The plot of $\log q_e$ vs. $\log C_e$ will obtain straight line with slope of $1/n$ and intercept of $\log K_F$. The K_F and n values were calculated from the intercept and slope of the plot represented in Table 1. The value of R^2 for Freundlich adsorption isotherm is 0.977 which is less

than Langmuir isotherm shown in Fig.9. The result shows that the system is not heterogeneous.

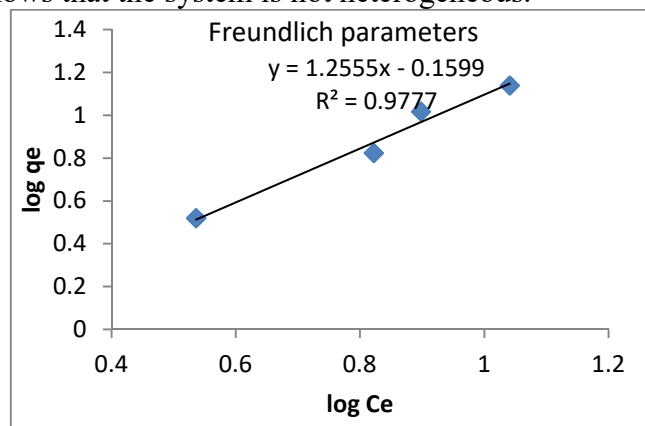


Fig 9. Shows Freundlich adsorption parameters.

Table 1. Adsorption isotherm parameter for the adsorption of MO dye

Freundlich parameters				
pH	1/n	n	K_F (mg/g)	R^2
5	1.255	0.796	1.442	0.977

Langmuir Isotherm

The Langmuir adsorption model is based on the assumption that the maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. The equation is given as

$$\frac{1}{q_e} = \frac{1}{q_m b C_e} + \frac{1}{q_m} \quad (5)$$

In equation (5) q_e represents the amount of dye adsorbed (mg/g), C_e is the equilibrium concentration of the adsorbate (mgL^{-1}), and q_m and b are Langmuir constants related to the maximum adsorption capacity (mg/g) and energy of adsorption (L/mg) [18].

According to equation (5), when the adsorption obeys the Langmuir equation, a plot of C_e/q_e versus C_e should be a straight line with a slope of $1/q_m b$ and intercept $1/q_m b$. This important characteristic of the Langmuir isotherm can be expressed in terms of a dimensionless factor, R_L .

$$R_L = 1/1 + bC_o \quad (6)$$

The R_L values indicate the type of adsorption as either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$), or irreversible ($R_L = 0$).

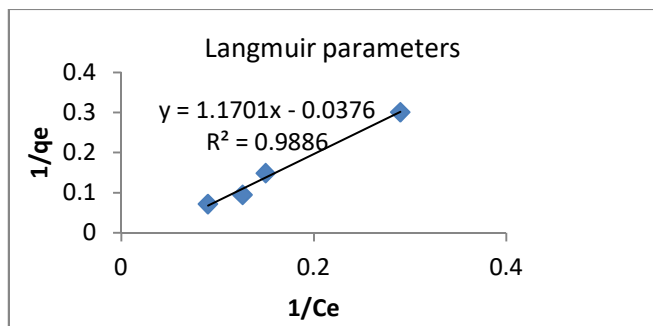


Fig 10. Shows Langmuir adsorption parameters.

Table 2. Adsorption isotherm parameter for the adsorption of MO dye

Langmuir parameters				
pH	q_m (mg/g)	b(L/mg)	R_L	R^2
5	27.02	0.0316	0.2834	0.988

Fig. 10 represents the equilibrium dye isotherm of Methyl Orange(MO) on activated carbon. While increasing the concentration of methyl orange the adsorption equilibrium also increases along with dye adsorption. The adsorption was found linear over the entire concentration range studied with good correlation coefficient ($R^2=0.988$). The result shows that correlations fit in well into the Langmuir isotherm relative to the ones obtained from Frerundlich model. Since Langmuir equation assumes that the surface is homogeneous.

Kinetic studies of adsorption

The adsorption kinetic study is quite significant as it describes the solute uptake rate which in turn controls the residence time of adsorbate uptake at the solid-solution interface. The dye concentration and adsorbent dose are the important parameter because they determine capacity of adsorbent for a given initial concentration of dye solution. The mechanism of adsorption is generally considered to involve three steps, one or any combination of which can be the rate-controlling mechanism: mass transfer across the external boundary layer film by of liquid surrounding the outside of the particle; adsorption at a site on the surface (internal or external) and the energy will depend on the binding process (physical or chemical); this step is often assumed to be extremely rapid; diffusion of the adsorbate molecules to an adsorption site either by a pore diffusion process through the liquid filled pores or by a solid-surface diffusion mechanism

[19], [20]. The kinetics of adsorption of Methyl Orange on activated carbon were analysed using pseudo first order (Lagergren), Pseudo second order kinetic models. The conformity between experimental data and the model predicted values were expressed by correlation coefficient (R^2). A relatively high R^2 value indicates that the model successfully describes the kinetics of Methyl Orange adsorption. The dye removal from the solution is through the mechanism of adsorption.

The pseudo-first-order kinetic model assumes that adsorption rate is proportional to the difference between q_e and q_t . The pseudo-second-order equation is based on the adsorption capacity of the solid phase. The pseudo-first-order kinetic equation

$$\ln q_e - q_t = \ln q_e - k_1 t \quad (7)$$

The pseudo-second-order kinetic model

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad (8)$$

where q_e (mg g^{-1}) is the adsorption capacity at equilibrium, and q_t (mg g^{-1}) is the adsorption amount at time t (min), k_1 ($\text{g mg}^{-1} \text{min}^{-1}$) and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) are the rate constant of pseudo-first-order and pseudo-second-order, respectively [21].

Values of k_1 at ambient temperature were calculated from the plots of $\log (q_e - q_t)$ versus t for different initial concentrations of MO. Values of k_2 at ambient temperature were calculated from the plots of t/q_t versus t [22]. The experimental data were fitted according to Lagergren pseudo first- order equation (7) and pseudo second-order equation (8) respectively.

We obtained a good linear plot for pseudo second-order reaction model (Fig.11) with a correlation coefficient R^2 of 0.986 indicated that the adsorption rates of the MO dye onto activated carbon can be more appropriately described using pseudo second-order equation. The experimental result clearly showed that the activated carbon of cassia alata as an efficient adsorbent for the MO dye removal from aqueous solution [23].

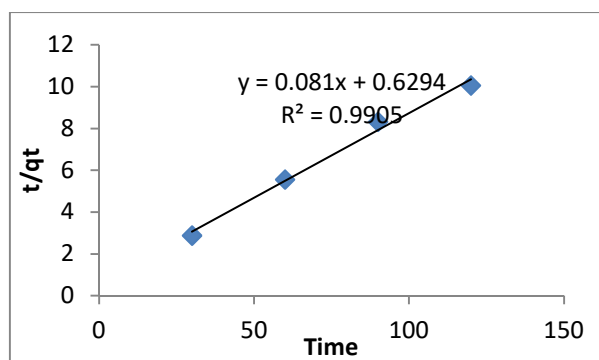


Fig.11, Pseudo-second-order kinetics plot for the adsorption of MO by CA-AC

Table 3. Pseudo Second order values

Pseudo second order			
qe (Experimental)	qe (Calulated)	K ₂	R ²
10.82	12.34	0.010	0.990

CONCLUSION

Activated carbon prepared from Cassia alata seeds, an agricultural solid waste and then used as a cheap adsorbent for the removal of Methyl Orange from aqueous solution. The parameters like contact time, pH, adsorbent dosage, and initial concentration of methyl orange were evaluated on sorption process. Effective removal of MO occurred at pH 5 and increase in concentration decreased the percentage of removal, and the adsorbent dose 0.3g the suitable temperature is 40°C with the contact time 40 min, obeys Langmuir adsorption isotherm, and follows second order kinetics. From this observations it an efficient adsorbent to remove MO and also eco friendly.

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