

Adsorption of Acid Red 18 from Aqueous Solution onto Activated Carbon Prepared from *Murraya koenigii* (Curry Tree) Seeds

S. SURESH^{1*}, R. WILFRED SUGUMAR² and T. MAIYALAGAN³

¹Department of Chemistry, Panimalar Institute of Technology, Chennai-602 103, India

²Department of Chemistry, Madras Christian College, Chennai-600 059, India

³South African Institute for Advanced Materials Chemistry, University of the Western Cape, Bellville 7535, Cape Town, South Africa

*Corresponding author: E-mail: avitsureshindia@gmail.com

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This paper focuses on the adsorption of acid red 18 onto low cost adsorbent prepared from *Murraya koenigii* seeds (MKS) from aqueous solutions at room temperature (25 °C). Batch experiments were conducted and the effect of different process parameters such as adsorbent dosage and initial dye concentration were studied. The adsorption isotherm data was analyzed using Langmuir, Freundlich and Temkin isotherm models. The equilibrium data fits well with Langmuir adsorption isotherm and the monolayer adsorption capacity was found to be 53.19 mg/g. The adsorption kinetics was studied using pseudo-first order and pseudo-second order models. The rate of adsorption was found to conform to pseudo-second order kinetics with a good correlation ($R^2 > 0.99$). The activated carbon prepared was characterized by SEM and FT-IR spectra. The results prove that *Murraya koenigii* seeds is a good low cost adsorbent for the removal of acid red 18 from aqueous solution.

Key Words: Activated carbon, Adsorption, Dye removal, Equilibrium, Kinetics.

INTRODUCTION

Dyes are widely used in textile, paper, leather, coir pith, carpet and cosmetic industries. The textile industry is one of the largest producers of industrial waste water. The effluent released from these industries, if not treated properly, is a major environmental concern. Effluents from the textile industry are highly coloured and disposal of this coloured water into the receiving water body causes damage to aquatic life and human beings^{1,2}. A lot of synthetic dyestuffs used in textile industry were found to be toxic, carcinogenic and even mutagenic³. Thus the effluents need to be treated properly before being released in to the environment.

There are several treatment methods like photo degradation⁴, coagulation, flocculation⁵ and electrochemical oxidation^{6,7} available for the treatment of water containing dye effluents. Among these methods, adsorption⁸ is one of the most effective processes used for the removal of dye from waste water.

Considerable work has been done to remove toxic dyes from the effluents using low cost adsorbents. Some of the materials used are apricot stone⁹, sugarcane bagasse¹⁰, rubber (*Hevea brasiliensis*) seed coat¹¹, neem (*Azadirachta indica*) leaf powder¹², coir pith¹³, bamboo dust, coconut shell, groundnut shell, rice husk, straw¹⁴, orange peel¹⁵, sunflower seed

hulls¹⁶, agricultural wastes¹⁷ etc. The present work is to investigate the adsorption phenomenon using *Murraya koenigii* seed as an adsorbent. The seed of *Murraya koenigii* or as it is commonly known as curry tree seed is abundantly available in south India and hence it is used as an adsorbent material for the removal of acid red 18 from aqueous solution.

EXPERIMENTAL

In this study a commercially available textile dyestuff namely acid red 18 is used as adsorbate. The molecular structure and properties of acid red 18 is shown in Fig. 1 and Table-1, respectively. The dye is widely used in textile industries. The selected dyestuff is a dye contaminant in the discharged effluents commonly used in dye houses.

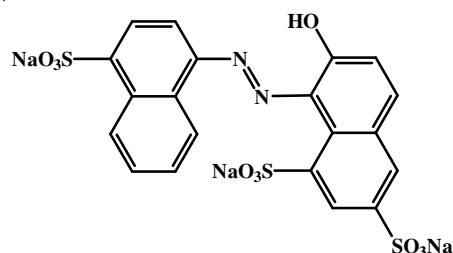


Fig. 1. Molecular structure of acid red 18

| TABLE-1 PROPERTIES OF ACID RED 18 | |
|--------------------------------------|--------------------------------|
| Parameters | Value |
| Chemical name | Acid Red 18 |
| CAS number | 2611-82-7 |
| Formula weight | 604.47 |
| Ionization | Basic |
| Molecular formula | $C_{20}H_{11}N_2O_{10}S_3Na_3$ |
| Chromophore | Monoazo |
| Maximum wavelength | 507 nm |

Preparation of activated carbon: Activated carbon used in this study was prepared by chemical activation method. The *Murraya koenigii* seeds (MKS) was washed several times to remove all dirt, dust and other surface impurities. The washed seeds were dried for 24 h. The dried seed was then soaked in 18 N H_2SO_4 (sulphuric acid) in the ratio 1:2 (w/v) and kept in a muffle furnace for 12 h at 120 °C. The carbonized material was finally crushed, sieved, dried and stored in plastic containers.

Characterization: The surface morphology of the activated carbon prepared from *Murraya koenigii* seeds was studied before and after dye adsorption by using scanning electron microscopy (Hitachi S3000N). The electronic structure of carbon samples were analyzed using FT-IR (Perkin-Elmer) spectrometer. The measurements were carried out in the range of 4000-450 cm^{-1} . Carbon samples (0.33 wt %) were stirred with dry KBr (Merck, spectroscopy grade) and then pressed to form appropriate tablets.

Batch adsorption experiments: The effect of various process parameters like adsorbent dosage, pH and initial dye concentration for the removal of acid red 18, were carried out in batch experiments in an orbital shaker at a constant speed of 160 rpm. The adsorption isotherm studies were carried out by agitating 50 mL of dye solution of various concentrations and fixed amount of adsorbent. After agitation, the dye solutions were separated from the adsorbent by centrifuging (Research Centrifuge, Remi Scientific works, Mumbai) for 5 min. The dye removal was determined spectrophotometrically (ELICO: SL 207) by monitoring the absorbance changes at the wavelength of maximum absorbance (507 nm). The effect of adsorbent dosage was studied with different adsorbent doses (25-150 mg). Kinetic experiments were carried out using a mechanical stirrer in the concentration range of 60-100 mg/L. The percentage dye removal was accessed using the equation

$$\text{Percentage of dye removal} = \frac{(C_i - C_f)}{C_i} \times 100 \quad (1)$$

The amount adsorbed at equilibrium q_e (mg/g) was calculated by

$$\text{Amount adsorbed } (q_e) = \frac{(C_i - C_f)V}{M} \quad (2)$$

where C_i and C_f are the liquid phase concentrations of the dye at initial and equilibrium concentrations (mg/L), respectively, M = mass (g) of adsorbent and V = volume of dye solution (L) Langmuir and Freundlich isotherms were employed to study the adsorption capacity of the adsorbent.

RESULTS AND DISCUSSION

Characterization of the adsorbent: Morphological study by SEM of the adsorbent shown in Fig. 2(a-b) revealed that

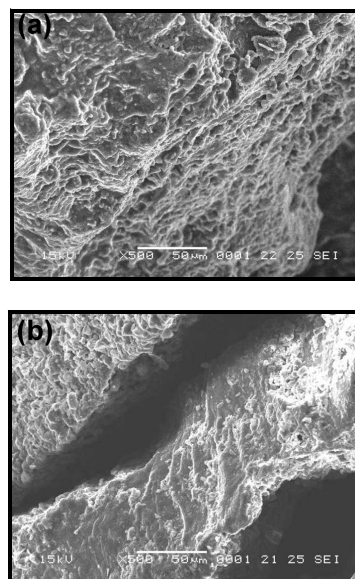


Fig. 2. SEM micrograph of prepared activated carbon (a) before dye adsorption (b) After dye adsorption

there are marked changes on the surface morphology of the adsorbent before and after dye adsorption. From the SEM pictures, it is obvious that the porous nature¹⁸ of the adsorbent would have increased the possibility for the dye to be trapped on the adsorbent.

The functional groups present on the adsorbent were confirmed by FT-IR spectra (Fig. 3). The adsorbent gives a number of absorption peaks. The peak at 3387.5 cm^{-1} represented bonded hydroxyl groups. A weak alkyl C-H stretching band is observed at 2931 cm^{-1} . The peak at 1724.5 cm^{-1} corresponds to C=O stretching. The peak at 1621.5 and 1231 cm^{-1} corresponds to C=C stretching and $-SO_3$ stretching, respectively. The peak observed at 1054.5 and 885.5 cm^{-1} could be assigned to C-O stretching vibration and C-H out of plane bending vibration. The adsorbent exhibits reasonable anionic exchange capacity due to the presence of polar functional groups on the adsorbent.

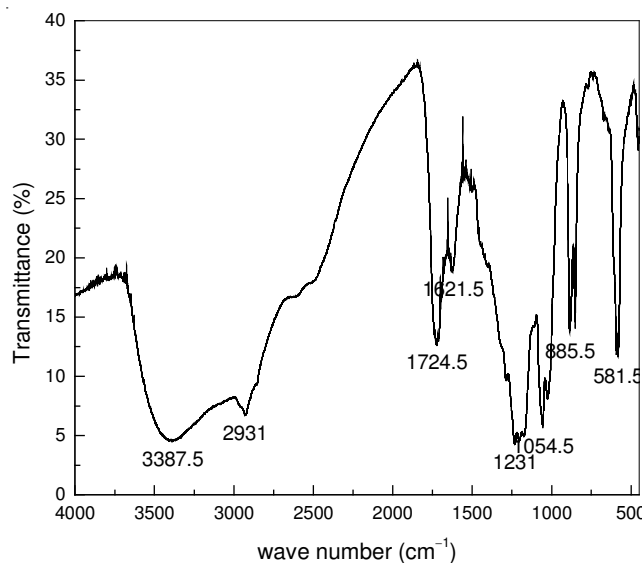


Fig. 3. FT-IR Spectra of the activated carbon prepared from *Murraya koenigii* seeds

Effect of adsorbent dosage: A fixed concentration of dye (40 mg/L) was taken and the adsorbent dosage was varied in the range of 25-150 mg at a constant volume (50 mL) keeping all other experimental conditions constant.

Fig. 4 shows that, an increase in adsorbent dosage increases the adsorption amount until a particular dosage at which adsorption becomes constant. The increase in adsorption following an increase in adsorbent dosage can be attributed to the availability of more adsorption sites⁸. However, the amount adsorbed per unit mass of the adsorbent decreases considerably. As the adsorption sites remain unsaturated during the adsorption process there is a decrease in unit adsorption with increasing dose of adsorbent¹⁶. When the adsorbent and the solute concentration are low then the sorption rate is low. On the other hand, a high adsorbent to solute concentration ratio increases the rate of adsorption on the adsorbent surface and hence there is a decrease in the solute concentration in solution.

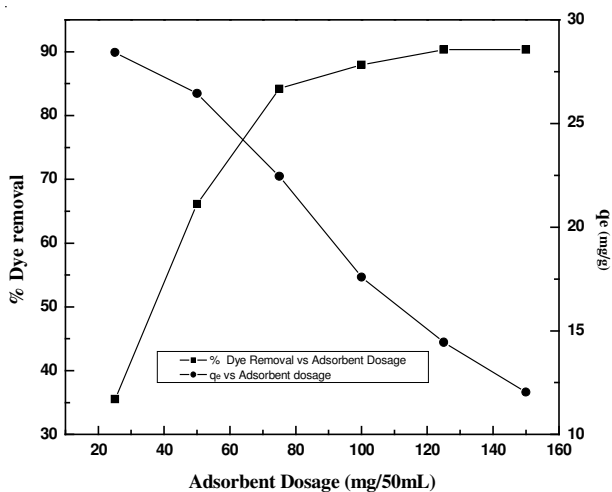


Fig. 4. Percentage of dye removal *versus* adsorbent dosage ($C_0 = 40$ ppm; $V = 0.05$ L; $t = 8$ h)

Effect of initial dye concentration (C_i): The effect of initial dye concentration was studied by using a fixed adsorbent dosage (50 mg) for different initial dye concentrations (40, 50, 60, 70, 80, 100 and 120 mg/L).

From Fig. 5, it can be observed that the percentage removal of dye decreases with the increase in initial concentration of dye. This is due to the saturation of the adsorption sites at higher concentrations. It is also observed that the actual amount of dye adsorbed increases with the increase in initial dye concentration (C_i). An increase in C_i increases the interactions between the adsorbent and adsorbate. Therefore there is an increase in the rate of adsorption of dye.

Adsorption isotherms: In accessing the equilibrium nature of adsorption of the adsorbent, three well known isotherms namely Langmuir, Freundlich and Temkin were selected for the present study. An adsorption isotherm describes the relationship between the mass of the adsorbate that is adsorbed per unit weight on the adsorbent and the concentration of dissolved adsorbate in the liquid phase at equilibrium.

Langmuir adsorption isotherm: The Langmuir adsorption isotherm is based on the assumption¹⁹ that an adsorbent adsorbs the dye at specific homogeneous sites and once that site is occupied, no further adsorption takes place at that site.

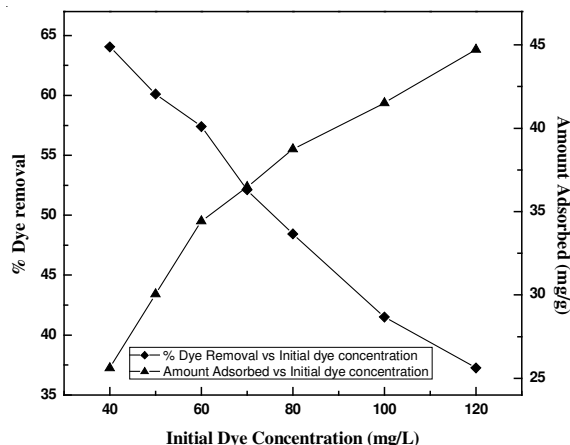


Fig. 5. Percentage dye removal *versus* initial dye concentration ($m = 0.05$ g; $V = 0.05$ L; $t = 8$ h)

Theoretically, the adsorbent has a finite capacity to absorb the adsorbate and hence there is a limit beyond which no further adsorption can occur²⁰. The monolayer capacity can be represented by the expression:

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

The linear form of the above equation is represented as:

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \quad (4)$$

where K_L is related to the free energy of adsorption (L/mg) and Q_m is maximum adsorption capacity. The values of Q_m and K_L were calculated from the slope and intercept of the linear plot (Fig. 6). The equilibrium concentration and hence the amount of dye adsorbed were calculated by varying the dye concentration between 40 and 120 mg/L with a fixed adsorbent dose and all other experimental conditions kept constant.

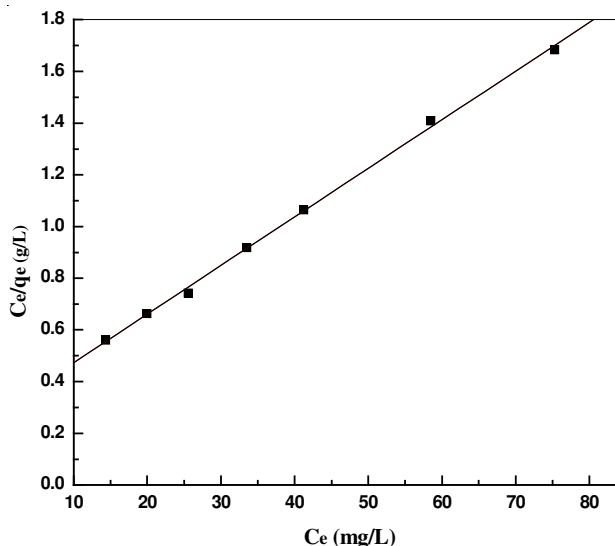


Fig. 6. Langmuir isotherm for acid red 18 adsorption onto *Murraya koenigii* seeds

The isotherm constants for the Langmuir isotherm under study were calculated from the linear form of the model and the correlation coefficients are given in Table-2.

| TABLE-2 LANGMUIR, FREUNDLICH AND TEMKIN CONSTANTS FOR THE ADSORPTION OF ACID RED 18 ONTO <i>Murraya koenigii</i> SEEDS | |
|---|--------|
| Adsorbent | Values |
| Langmuir | |
| q_m (mg/g) | 53.19 |
| K_L (L/mg) | 0.066 |
| R^2 | 0.9992 |
| Freundlich | |
| K_F (mg/g) | 11.54 |
| n | 3.12 |
| R^2 | 0.9801 |
| Temkin | |
| K_T (L/mg) | 0.762 |
| B_1 | 11.122 |
| R^2 | 0.9921 |

An essential characteristic of Langmuir isotherm can be expressed in terms of a dimensionless constant called the equilibrium parameter²¹.

$$R_L = \frac{1}{(1 + K_L C_0)} \times C_0 \quad (5)$$

The value of R_L indicates the type of isotherm to be favourable ($0 < R_L < 1$), linear ($R_L = 1$), unfavourable ($R_L > 1$) or irreversible $R_L = 0$, where K_L is the Langmuir constant and C_0 is highest initial dye concentration (mg/L). The value of R_L is found to be in the range of 0.2747 to 0.1121 for a dye concentration of 40-120 mg/L indicating favourable adsorption of acid red 18 onto MKS.

Freundlich adsorption isotherm: Freundlich isotherm is an empirical equation employed to describe heterogeneous systems. The Freundlich equation is commonly given by:

$$q_e = K_F C_e^{1/n} \quad (6)$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g), C_e is the equilibrium concentration of solute in the bulk solution (mg/L), K_F and n are Freundlich constants indicating the relative adsorption capacity and sorption intensity of the adsorbents (mg/g). A linear form of the Freundlich expression can be obtained by taking logarithms of the non-linear form and can be written as

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \quad (7)$$

The Freundlich constants K_F and n can be calculated from the slope and intercept of the linear plot with $\log q_e$ versus $\log C_e$ (Fig. 7).

The Freundlich model assumes that the adsorption of the dye occurs on a heterogeneous surface by multilayer adsorption with the total adsorption increasing rapidly with an increase in concentration.

According to Treybal²² it has been shown using mathematical calculations that 'n' values in the range of $0 < n > 1$ represents favourable adsorption. The isotherm constants K_F and n were calculated from the linear form of the model and the value of K_F , n and correlation coefficients are given in the Table-2.

Temkin adsorption isotherm: The effect of indirect sorbate/adsorbate interactions on adsorption isotherms is considered

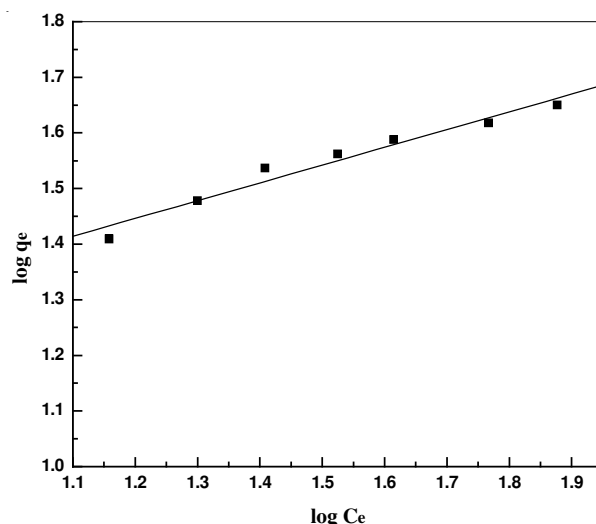


Fig. 7. Freundlich isotherm for acid red 18 adsorption onto MKS

by Temkin and Pyzher and it is suggested that the heat of adsorption and the interactions of the molecules decrease linearly with coverage²³. The adsorption is characterized by a uniform distribution of binding energy. The Temkin equation is given as

$$q_e = \frac{RT}{b} \ln(K_T C_e) \quad (8)$$

or, the above equation can be simplified in the linear form as

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (9)$$

where $B_1 = RT/b$ and K_T is the Temkin isotherm constant (L/g) where these constants can be calculated from a plot of q_e versus $\ln C_e$ (Fig. 8) and are presented in Table-2.

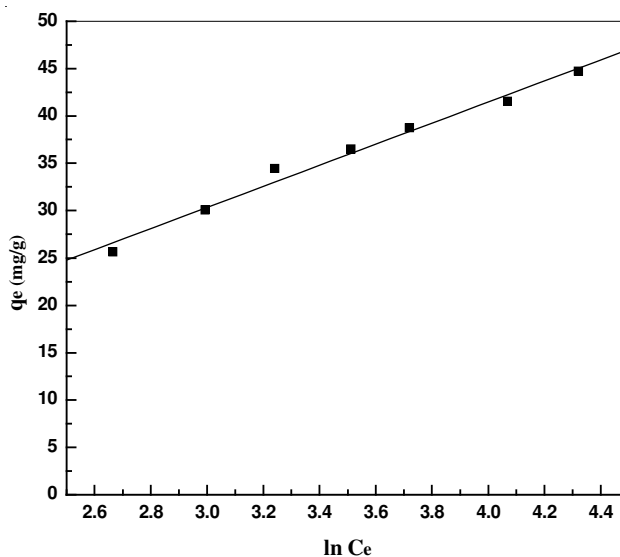


Fig. 8. Temkin isotherm for acid red 18 adsorption onto *Murraya koenigii* seeds

The linear form of the Temkin equation is used to analyze the adsorption data and it is observed that the Temkin isotherm fitted in comparatively well when compared with the Freundlich isotherm. The R^2 value (0.9921) is also good when compared to the Freundlich isotherm which proves the applicability of Temkin isotherm for the adsorption of acid red 18 on to *Murraya koenigii* seeds.

The Langmuir, Freundlich and Temkin curve for acid red 18 adsorption onto *Murraya koenigii* seeds along with the experimental data are given in Fig. 9. It is clear that the experimental data fit closely with predicted data for Langmuir adsorption isotherm with R^2 values fitting the following series; Langmuir > Freundlich > Temkin, for adsorption of acid red 18 on to *Murraya koenigii* seeds. The surface chemistry of the activated carbon is a key factor in interpreting the dye adsorption process²⁴. The presence of surface oxygen groups on the carbon prepared from *Murraya koenigii* seeds imparts a polar character to the activated carbon surface, that provides electrostatic attraction for the preferential adsorption of anionic dye molecules on the carbon surface and hence from the results obtained it is evident that *Murraya koenigii* seeds could be used as an effective low cost adsorbent for the removal of dye from aqueous solution.

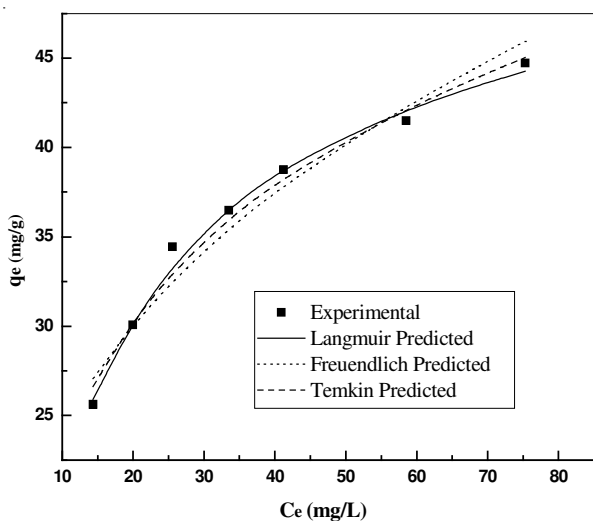


Fig. 9. Comparative fit of Langmuir, Freundlich and Temkin isotherm with the equilibrium data plotted as q_e versus C_e ($m = 0.05$ g; $V = 0.05$ L; $t = 8$ h)

Kinetics: Adsorption process rate can be explained by adsorption kinetics. It is observed that activated carbon prepared from *Murraya koenigii* seeds adsorb dyes effectively and the process attains equilibrium slowly with the rate of adsorption influenced by the presence of functional groups such as carboxylic, carbonyl, hydroxyl, ether, lactone *etc.*, on the carbon surface that tends to generate different types of adsorbent-adsorbate interactions²⁵.

Pseudo-first order kinetics: A simple kinetic analysis of adsorption, the pseudo first order kinetics and its integrated form is given by

$$\log (q_e - q_t) = \log q_e - \frac{K_1}{2.303t} \quad (10)$$

where K_1 is the first order rate constant that is calculated from the slope of the plot of $\log (q_e - q_t)$ versus time (Fig. 10). A plot of $\log (q_e - q_t)$ versus time enables calculation of the rate constant K_1 and from the slope and intercept of the plot, $q_e^{(pred)}$ can be calculated. The pseudo first order model data do not fall on the straight line for different initial concentrations studied, it is also observed that the predicted q_e value and the experimental q_e values do not coincide and hence pseudo-first order model is not applicable.

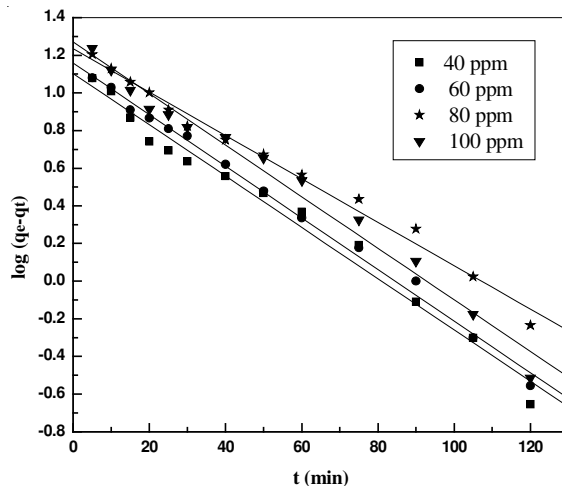


Fig. 10. Pseudo first order kinetics plot at four different initial concentration of acid red 18 onto *Murraya koenigii* seeds

Pseudo-second order kinetics: The pseudo second order model is given by the equation

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_t} \quad (11)$$

A plot of t/q_t versus t (Fig. 11) enables calculation of the rate constant K_2 which in turn is used to calculate the initial sorption rate h as follows:

$$h = K_2 q_e^2 \quad (12)$$

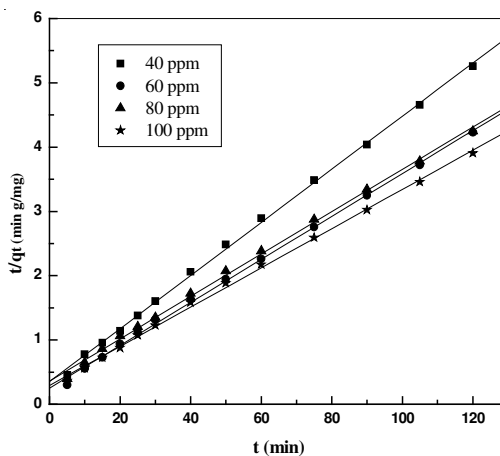


Fig. 11. Pseudo second order kinetics plot at four different initial concentration of acid red 18 onto *Murraya koenigii* seeds

The q_e values calculated from the pseudo-second order model system are in good agreement with the experimental q_e values. It is observed that the K_2 value decreases with increasing dye concentration and then increases. The correlation coefficient (R^2) of the linear plot is very high (> 0.99) for all the concentrations studied. This indicates that the adsorption follows the second order model and the adsorption process is a chemisorption process. The values of kinetic constants and q_e of acid red 18 adsorption onto *Murraya koenigii* seeds are given in Table-3.

Table-4 gives the adsorption capacity of some of the low cost adsorbents used for adsorption of dyes. From the Table-4, it is seen that the adsorption ability of the carbon prepared

TABLE-3
KINETIC MODEL VALUES FOR THE ADSORPTION OF ACID RED 18 ON TO ACTIVATED *Murraya koenigii* SEEDS (MKS)

| Concentration (ppm) | $q_{e(\text{exp})}$ (mg/g) | Pseudo first order values | | | Pseudo second order values | | | |
|---------------------|----------------------------|-----------------------------|-----------------------------|--------|-----------------------------|--------------------------------------|------|--------|
| | | $q_{e(\text{calc})}$ (mg/g) | K_1 (min^{-1}) | R^2 | $q_{e(\text{calc})}$ (mg/g) | K_2 ($\text{g}/(\text{mg min})$) | h | R^2 |
| 40 | 23.06 | 12.67 | 0.03132 | 0.9823 | 24.21 | 0.004891 | 2.86 | 0.9989 |
| 60 | 28.71 | 14.41 | 0.03155 | 0.9950 | 29.94 | 0.004520 | 3.76 | 0.9987 |
| 80 | 28.86 | 17.15 | 0.02648 | 0.9888 | 30.39 | 0.003014 | 2.78 | 0.9979 |
| 100 | 31.05 | 18.66 | 0.03155 | 0.9818 | 32.78 | 0.003212 | 3.45 | 0.9989 |

TABLE-4
ADSORPTION CAPACITIES OF DIFFERENT LOW COST ADSORBENTS FOR DYE REMOVAL FROM AQUEOUS SOLUTION

| Adsorbent | Dye material | Adsorption capacity (mg/g) | Reference number |
|-------------------------------|----------------|----------------------------|------------------|
| Orange peel | Acid violet 17 | 19.88 | 15 |
| Coir pith | Acid violet | 8.06 | 26 |
| Sugarcane bagasse | Acid orange 10 | 5.78 | 27 |
| Rice husk | Acid blue | 50.00 | 28 |
| Egyptian bagasse pith | Acid blue 25 | 17.50 | 29 |
| Egyptian bagasse pith | Acid red 114 | 20.00 | 29 |
| Wood sawdust | Acid blue 25 | 5.99 | 30 |
| <i>Murraya koenigii</i> seeds | Acid red 18 | 53.19 | Present study |

from *Murraya koenigii* seeds is comparatively good when compared with some of the low cost adsorbents already reported in the literature for the adsorption of acid dyes from aqueous solution. The adsorption capacity of the adsorbent (q_{max}) prepared suggests that acid red 18 is comparatively easily adsorbed by the activated carbon used in the present study.

Conclusion

The adsorption isotherm was best defined by the Langmuir isotherm model and the adsorption capacity found to be 53.19 mg/g for the prepared activated carbon. The adsorption process follows pseudo-second order model with good correlation. The R_L value (from Langmuir Isotherm Table-2) between 0 and 1 shows favourable adsorption. The material used in this study is an agricultural waste product, economical to use as an alternative to costlier adsorbents used for dye removal in wastewater treatment processes.

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