

Adsorption of Methylene Blue From Waste Water Using Activated Carbon Prepared from Copper Pod

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ABSTRACT: The ability of activated carbon produced from copper pod to remove methylene blue from aqueous solution was investigated. The effects of different variables such as initial dye concentration, adsorbent dosage, pH, contact time and temperature were studied. The results obtained showed that the adsorption of methylene blue is dependent on adsorbent dosage, pH and contact time. The optimum adsorbent dosage, pH, contact time and temperature were found to be 1g, pH 7, 60 min and 30 °C, respectively. Adsorption equilibrium data of aqueous solutions were analysed using Langmuir and Freundlich isotherms. Adsorption kinetics data of aqueous solutions were studied using pseudo-first-order, pseudo-second-order and intraparticle diffusion models. The morphology was investigated by scanning electron microscope and X-ray diffraction data. The equilibrium data were best represented by Langmuir isotherm. From adsorption kinetics data, it was found that the adsorption of methylene blue followed pseudo-second-order kinetics. The results indicated that the activated carbon prepared from copper pod could be used as a low-cost alternative adsorbent for the removal of methylene blue from aqueous solution.

Keywords: Copper pod·Methylene blue·Adsorption·Isotherms·Kinetics

I. INTRODUCTION

Water is the most abundant and the most useful element for the existence of human beings, animals and plants. One of the major environmental problems is wastewater pollution. Wastewater from textile industry has high biological oxygen demand, increased chemical oxygen demand and changed pH [1]. The release of wastewater from textile industry causes several problems such as altered colour and taste of the water [2, 3]. Dyes are used in various industries such as textiles, plastics, leather, paper and pulp, cosmetics and food industries to colour their product [4, 5]. Discharge of these dyes into the water bodies not only poses certain hazards but also inhibits sunlight penetration into the stream and affects aquatic ecosystem [6, 7]. It is necessary to remove dyes from an industrial effluent before it is discharged into nearby water bodies. Several methods are used for removing dyes from wastewater including coagulation and flocculation, oxidation, electrocoagulation and membrane separation, ion exchange, electrochemical destruction, precipitation, electroflotation and irradiation [8, 9]. However, all these treatment methods are not widely used because of their high cost. Hence, adsorption process is the most widely used technique for the removal of dyes from wastewater.

II. MATERIALS AND METHODS

2.1. Preparation Of Adsorbent

Copper pod (perunkonrai in Tamil, konda chinta or pachha sunkesula in Telugu, radhachura in Bengali) is a species of *Peltophorum* native to tropical southeastern Asia, Australia, India, Indonesia, Malaysia, Singapore, Sri Lanka, Thailand, Vietnam and so on. Flowering occurs from March to May and a second flush of flowers may occur from September to November. *Peltophorum pterocarpum* is a fast growing tree, attaining a height of 9m in 3 years and flowering when around 4 years. It is used as fuel wood and is a widely appreciated shade tree.

Copper pod-activated carbon was used as an adsorbent for dye removal in this study. The copper pod fruit was first peeled off to obtain the outer skin of the fruit and then the inner fleshy layer was removed. The peel was washed with ordinary tap water to remove any dirt or sand. The washed materials were dried in sunlight to evaporate the moisture present in it. The dried materials were carbonized with 1:1 sulphuric acid. The charred material was filtered and washed with excess of water to remove the residual acid from the pores of

the carbon particles. The filtered material was kept in a muffle furnace at 600 °C for 30 min. The carbonized material was ground to fine powder and then filtered with a sieve of particle size 53 µm. The sieved adsorbent was kept in an airtight container for further adsorption studies.

2.2. Preparation Of Adsorbate

In this study, methylene blue was used as an adsorbate. It has a molecular weight of 320 and its molecular formula is C₁₆H₁₈N₃SCl. The structure of the dye is shown in Fig.1. A stock solution of methylene blue was prepared by dissolving 1 g methylene blue in 1 L distilled water to give a concentration of 1000mg/L. The working solutions were prepared by diluting the stock solution with distilled water to get the appropriate concentration. The concentration of the residual dye solution was measured using UV/visible spectrometer at a λ_{max} value of 663nm.

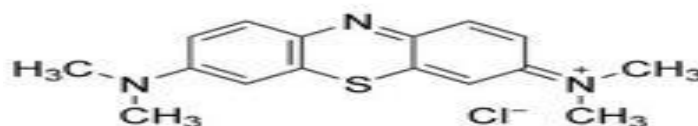


Fig. 1. Molecular Structure Of Methylene Blue

2.3. Effect of agitation time and initial dye concentration

The effect of agitation time and initial dye concentration was studied by agitating 100mg of the activated carbon with 50mL methylene blue solution in a mechanical shaker. After agitation, the solution was filtered and analysed using a digital spectrophotometer.

2.4. Effect of adsorbent dosage

The effect of adsorbent dosage was studied by preparing different dye concentrations of 10, 20, 30, 40 and 50 mg/L and different adsorbent dosages of 0.1–1g.

2.5. Effect of initial pH

The effect of pH on the amount adsorbed was studied by assessing various pH values of the solution, from 2 to 12. The pH of the solution was controlled using 0.1M HCl or 0.1M NaOH.

2.6. Effect of temperature

The effect of temperature on the amount of dye removal was studied for different temperatures (30, 40, 45, 50,55and 60 °C) for 60 min at a constant stirring speed.

2.7. Scanning electron micrograph study

The scanning electron microscope (SEM) image was used to investigate the nature and surface characteristics of the prepared activated carbon. The image was taken before the adsorption of methylene blue on the adsorbent.

2.8. X-ray diffraction measurement

The instrument was equipped at a voltage of 30 kV and a current of 30 mA with a Cu Kα radiation and scanning range of θ–2θ configuration. This X-ray diffraction (XRD) pattern was analysed to investigate the crystallographic structural changes during adsorption processes.

2.9. Langmuir isotherm model

The Langmuir model suggested that homogeneous monolayer adsorption onto a surface contains a finite number of adsorption sites. This model assumes that there are uniform energies of adsorption on the adsorbent surface and there is no transmigration of adsorbate in the plane of the surface.

The Langmuir model can be expressed as follows:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad (1)$$

where Q₀ Langmuir is the constant related to adsorption capacity (mg·g⁻¹),
 b is the Langmuir constant related to rate of adsorption(L·mg⁻¹),
 C_e is the concentration of dye solution at equilibrium (mg·L⁻¹) and
 q_e is the amount of dye adsorbed on the adsorbent at equilibrium (mg·g⁻¹).

Q_0 and b values can be calculated from the slope and the intercept of the straight line of the plot of $\frac{C_e}{q_e}$ versus C_e .

The essential characteristic of the Langmuir isotherm was expressed in terms of dimensionless equilibrium parameter R_L also called as the separation factor. It is expressed by the following equation:

$$R_L = \frac{1}{1+bc_0} \quad (2)$$

where b is the Langmuir constant ($L \cdot mg^{-1}$) and C_0 is the initial concentration of dye ($mg \cdot L^{-1}$). The value of R_L indicates the shape of the isotherm, and whether it is favourable ($0 < R_L < 1$), unfavourable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$).

2.10. Freundlich isotherm

The Freundlich isotherm is an empirical equation and deals with heterogeneous adsorption. The Freundlich isotherm equation is expressed as follows:

$$\log q_e = \log k_f + \left(\frac{1}{n}\right) \log C_e \quad (3)$$

where

q_e is the amount of dye adsorbed ($mg \cdot g^{-1}$),

C_e is the concentration of dye solution at equilibrium ($mg \cdot L^{-1}$),

k_f is the Freundlich constant related to the adsorption capacity of adsorbent,

n is the Freundlich constant related to the adsorption intensity, and

k_f and n values can be calculated from the slope of the straight line.

The values of k_f and n can be determined from the slope and intercept of plot of $\log q_e$ versus $\log C_e$. The slope of $1/n$ value ranges between 0 and 1. It is a measure of adsorption intensity (surface heterogeneity) becoming more heterogeneous as its value gets near zero. If $1/n$ value is below 1, a normal Langmuir isotherm occurs. If $1/n$ value is above 1, cooperative adsorption occurs.

2.10. Adsorption kinetics

The study of adsorption kinetics is an important characteristic in describing the efficiency of the adsorption process. The adsorption kinetics parameters are useful for the prediction of rate of adsorption and modelling the adsorption process. Various adsorption kinetics models have been used to analyse the kinetics of the adsorption process. The adsorption kinetics parameters were determined from the pseudo-first-order and second-order equations. Both the models are the most widely used for the adsorption of solutes from a liquid solution.

The pseudo-first-order kinetic model can be expressed by the following linear form of equation:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (4)$$

where q_e is the amount of dye removed at equilibrium ($mg \cdot g^{-1}$),

q_t is the amount of dye removed at time t ($mg \cdot g^{-1}$),

k_1 is the pseudo-first-order rate constant (min^{-1}).

k_1 and q_e values were determined from the slope and intercept of the plot of $\log(q_e - q_t)$ versus time t .

The pseudo-second-order kinetic model can be expressed by the following linear form of equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where q_e is the amount of dye removed at equilibrium ($mg \cdot g^{-1}$),

q_t is the amount of dye removed at time t ($mg \cdot g^{-1}$),

k_2 is the pseudo-second-order rate constant (min^{-1}).

A plot of $\frac{t}{q_t}$ against time t gives a straight line from which k_2 and q_e values can be determined from the slope and intercept of the plot, respectively. A comparison between the pseudo-first-order and the pseudo-second-order kinetic models was then done.

2.11. Intraparticle diffusion studies

Intraparticle diffusion is one of the important models used for adsorption kinetics. The Weber and Morris intraparticle diffusion model is expressed as follows:

$$q_t = k_d t^{1/2} \quad (6)$$

where q_t is the amount of dye adsorbed ($mg \cdot g^{-1}$) at time t , and k_d ($\frac{mg}{g \cdot min}$) is the rate constant for intraparticle diffusion. The k_d values were calculated from the slope of the plots of q_t versus $t^{1/2}$.

III. RESULTS AND DISCUSSION

3.1. Effect Of Contact Time And Initial Dye Concentration

The amount of methylene blue adsorbed by the activated carbon prepared from copper pod at different initial concentrations of 10 to 50 mg·L⁻¹ was studied at 30°C with 100 mg fixed carbon dosage. Figure 2 shows that the percentage of methylene blue removal is increased with an increase in contact time. This is due to high affinity between copper pod and methylene blue [10, 11]. After the amount of adsorption reaches a limiting value, no further adsorption takes place [12, 13].

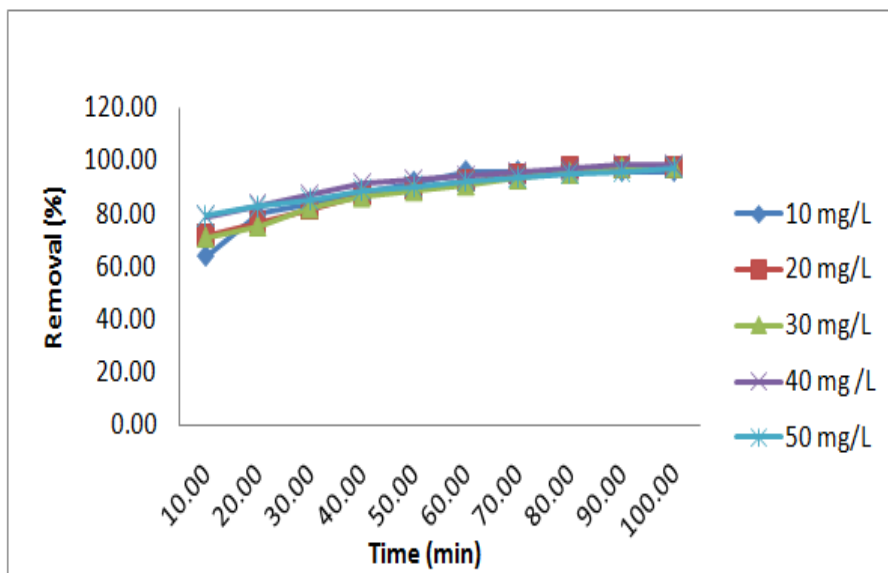


Fig. 2. Effect of initial concentrations on the adsorption of methylene blue onto copper pod.

3.2. Effect of carbon dosage on dye removal

The carbon dosage is an important parameter to determine the adsorption capacity of an adsorbent for initial concentration of the adsorbate at the operating conditions and is shown in Fig. 3. The increase in percentage of dye removal with an increase in carbon dosage from 0.1–1g is due to the increase in surface area and the number of active sites of the adsorbent. It was observed that the maximum dye removal capacity for activated carbon prepared from copper pod is 90% at normal pH and temperature conditions. The results indicated that the percentage of dye removal increases rapidly with an increase in the carbon dosage because of the availability of more binding sites [14, 15]. It was found that the complete dye removal from aqueous solution requires 1g of the prepared activated carbon from copper pod.

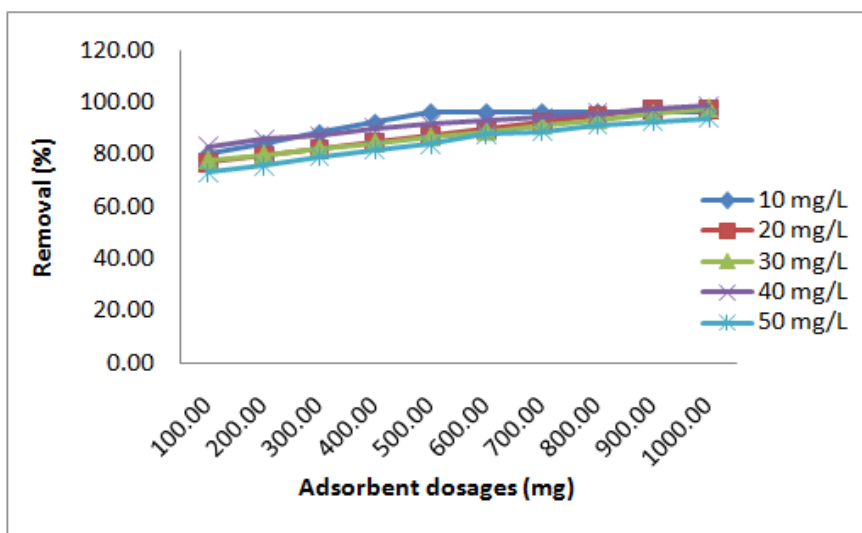


Fig. 3. Effect of adsorbent dosages on the adsorption of methylene blue onto copper pod.

3.3. Effect of pH

The pH of the solution is a very important factor in the adsorption process. The effect of pH on the adsorption of dye was explained over the pH range from 2 to 12. The effect of the pH on the adsorption of methylene blue onto copper pod is shown in Fig. 4. The adsorption of methylene blue increases with an increase in pH of the dye solution from 2 to 7 [16, 17] and decreases with an increase in pH of the dye solution from 8 to 12. The increase in the amount of dye removed with an increase in the pH of the dye solution was due to the nature and replacement of cationic groups of the dye on the surface of the activated carbon [18, 19]. The decrease in the amount of dye removed with an increase in the pH of the dye solution was due to the presence of excess OH^- ions that compete with the dye anions for the adsorption sites.

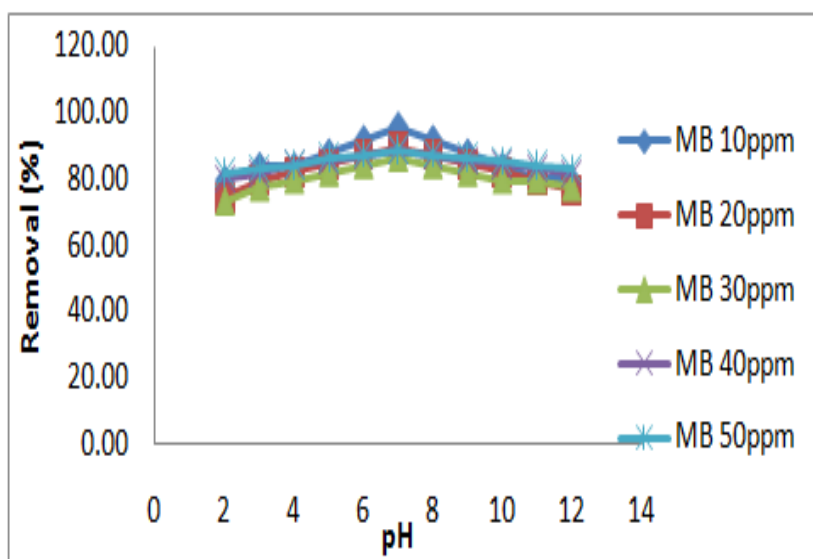


Fig. 4. Effect Of Ph On The Adsorption Of Methylene Blue Onto Copper Pod.

3.4. Effect of temperature

The effect of temperature on the adsorption was studied at different temperatures: 30, 40, 45, 50, 55 and 60°C. The percentage of dye removed at different temperature is shown in Fig. 5. It was observed that the percentage of dye removed by activated carbon prepared from copper pod decreases with increase in temperature. This is because higher temperature may decrease the adsorptive forces between the dye molecules and the active sites of the adsorbent surface [20, 21]. The optimum temperature at which maximum dye removal takes place is 30°C. The result also indicated that the adsorption process is exothermic in nature when temperature increases from 30 to 60°C and the initial dye concentration is 40mg·L⁻¹.

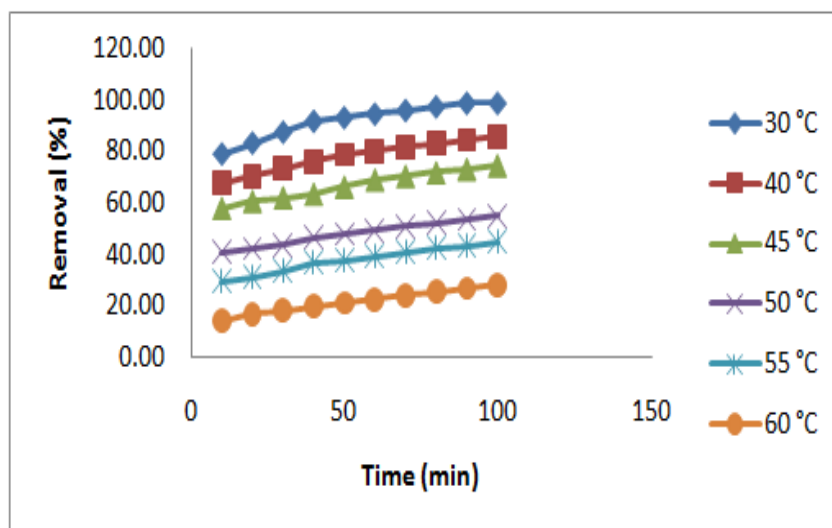


Fig. 5. Effect Of Temperature On The Adsorption Of Methylene Blue Onto Copper Pod.

3.5. Scanning electron micrograph study

The surface morphology of the adsorbent sample was analysed using SEM. Figure 6 shows SEM image of the adsorbent. This image shows the pores of different sizes and shapes of the adsorbent [22, 20]. The number of pores developed on the adsorbent plays a role in the adsorption process [23, 18]. This image also shows a clear view of the grains of the materials used as an adsorbent.

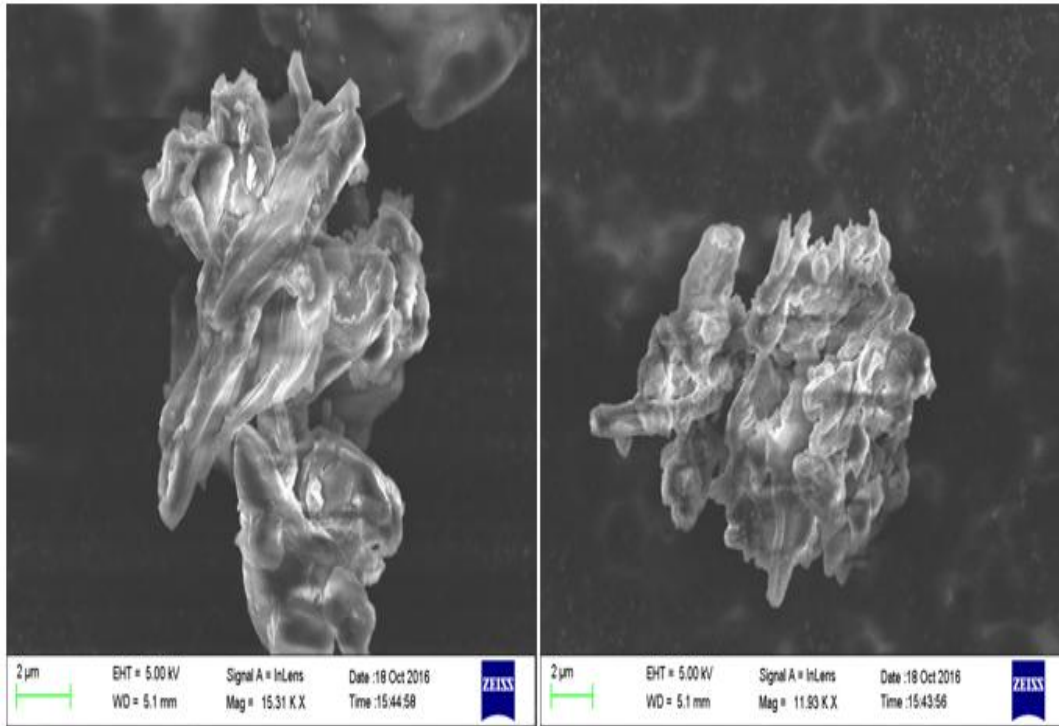
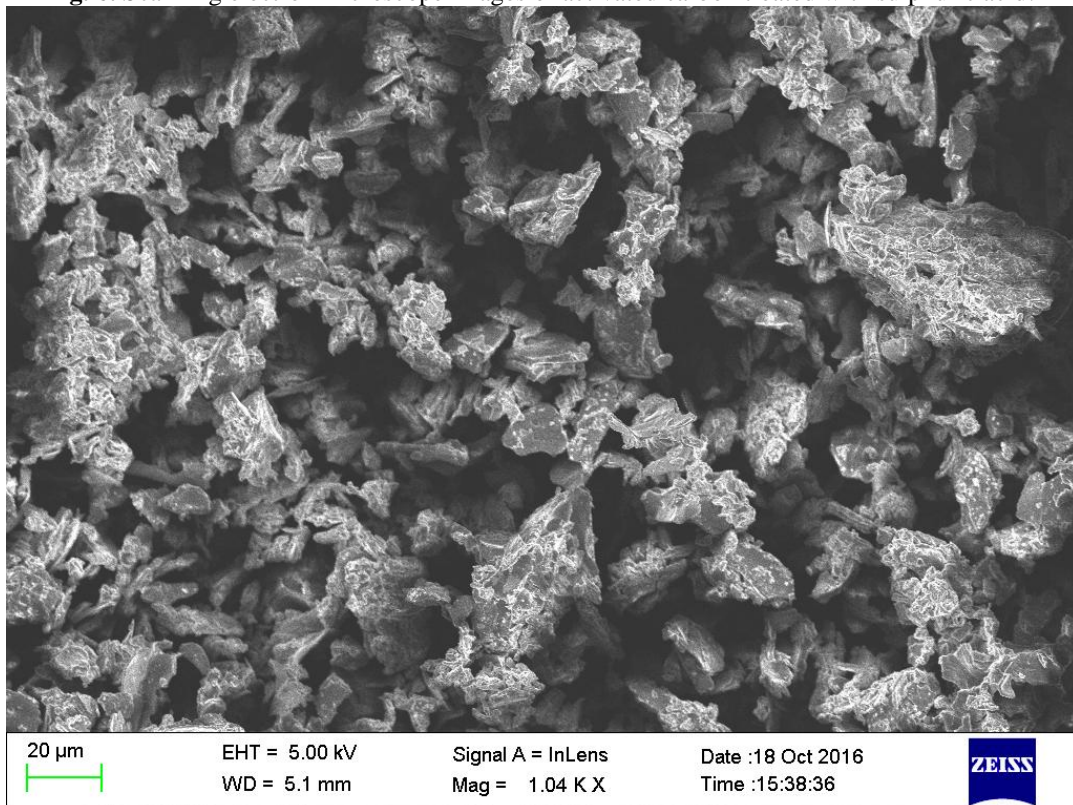


Fig. 6. Scanning electron microscope images of activated carbon treated with sulphuric acid.



3.6. Powder X-ray diffraction study

The powder XRD analysis of sample was carried out and the result is shown in Fig. 7. In activated carbon, a broad peak because of the reflections from the planes can be clearly seen. The broadness of the peak indicates the amorphous nature of the carbon sample [24, 16].

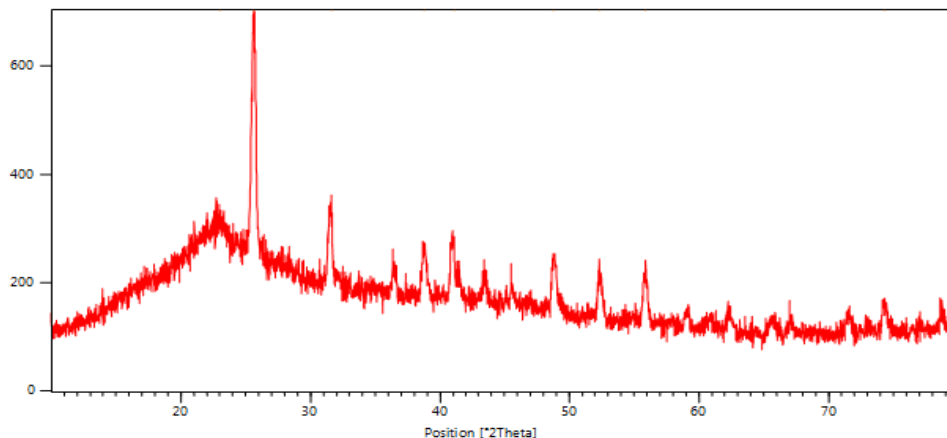


Fig. 7. X-ray diffraction pattern for prepared activated carbon.

Figure 7 shows XRD spectrum of the adsorbent. This spectrum clearly shows that the particle size is responsible for the broad peaks. This spectrum also indicates the presence of amorphous form of carbon, which is disorderly stacked up by carbon rings.

3.7. Adsorption isotherm

The adsorption isotherm is important for describing adsorption behaviour and the nature of the interaction between the adsorbate and adsorbent. Several isotherm equations were available to describe the experimental data for adsorption isotherms. In this study, adsorption isotherm was carried out on Langmuir and Freundlich models.

3.8. Langmuir isotherm

The values of adsorption capacity (Q_0) and energy of adsorption (b) can be determined from the slope and intercept of plots of C_e/q_e versus C_e . Q_0 and b values, which are given in Table 1. The linear plot of Fig.8 indicates that the adsorption obeys the Langmuir isotherm model. The values of Q_0 and b decrease with the increase in temperature [25, 26]. The experimental data indicate that the amount of dye adsorbed on the adsorbent decreased with the increase in adsorption temperature.

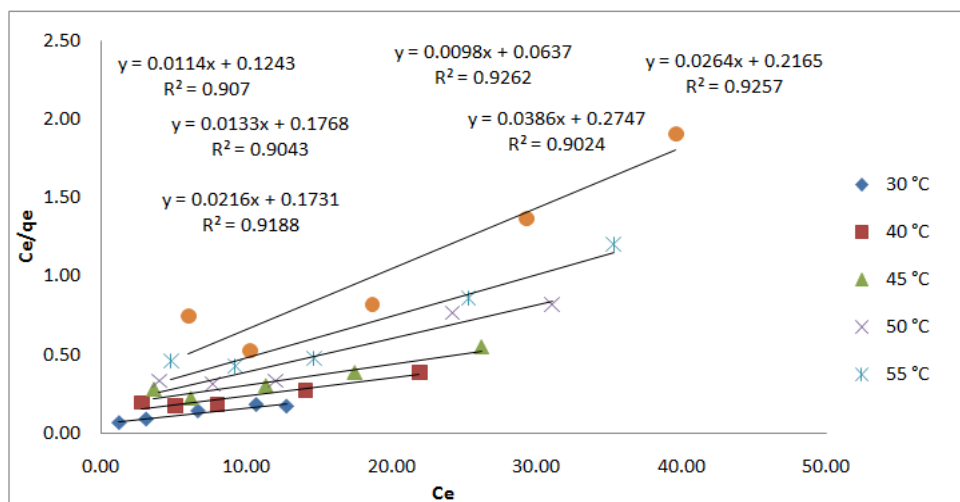


Fig. 8. Langmuir isotherm for the adsorption of methylene blue onto prepared activated carbon.

Table 1 Langmuir isotherm parameters

Temperature(D.)	Langmuir constants		
	R ²	Q ₀ (mg.g ⁻¹)	b (L.mg ⁻¹)
30	0.926	111.111	0.1428
40	0.907	90.9090	0.0887
45	0.904	76.9230	0.0738
50	0.918	47.6190	0.1213
55	0.925	38.4615	0.1203
60	0.902	26.3157	0.1386

Table 2 R_L values at various initial dye concentrations

Initial dye concentration (ppm)	R _L value					
	30°C	40°C	45°C	50°C	55°C	60°C
10	0.411	0.529	0.575	0.451	0.453	0.418
20	0.259	0.360	0.403	0.291	0.293	0.264
30	0.189	0.273	0.310	0.215	0.216	0.193
40	0.148	0.219	0.252	0.170	0.171	0.152
50	0.122	0.183	0.213	0.141	0.142	0.126

The linearity of the plots and also the high correlation coefficient values revealed that the adsorption obeys the Langmuir isotherm model with monolayer adsorption [27, 23]. This result clearly indicates that the adsorption of methylene blue on activated carbon prepared from copper pod takes place as monolayer adsorption on the surface of the adsorbent, homogenous in adsorption affinity and with constant adsorption energy. The constant (Q₀) is a measure of maximum adsorption capacity of the adsorbent under the experimental conditions [28, 29]. The constant (Q₀) decreased with the increase in temperature and the values were 111.1111, 90.9090, 76.9230, 47.6190, 38.4615 and 26.3157 mg.g⁻¹ for methylene blue adsorption at different temperatures, that is, at 30, 40, 45, 50, 55 and 60°C. The result indicates that the maximum adsorption occurs at 30°C.

The value of R_L indicates types of the isotherm to be linear(R_L =1),favourable (0<R_L<1), unfavourable(R_L>1), or irreversible(R_L=0).R_L values for the present experiment data fall between 0 and 1, which clearly indicates that the adsorption of methylene blue on activated carbon was favourable.

3.9. Freundlich isotherm

The values of Freundlich constants K and n can be calculated from the plot of log q_e versus log C_e, which gives a straight line with a slope of '1/n' and the result is shown in Fig. 9.The values are given in Table 3.

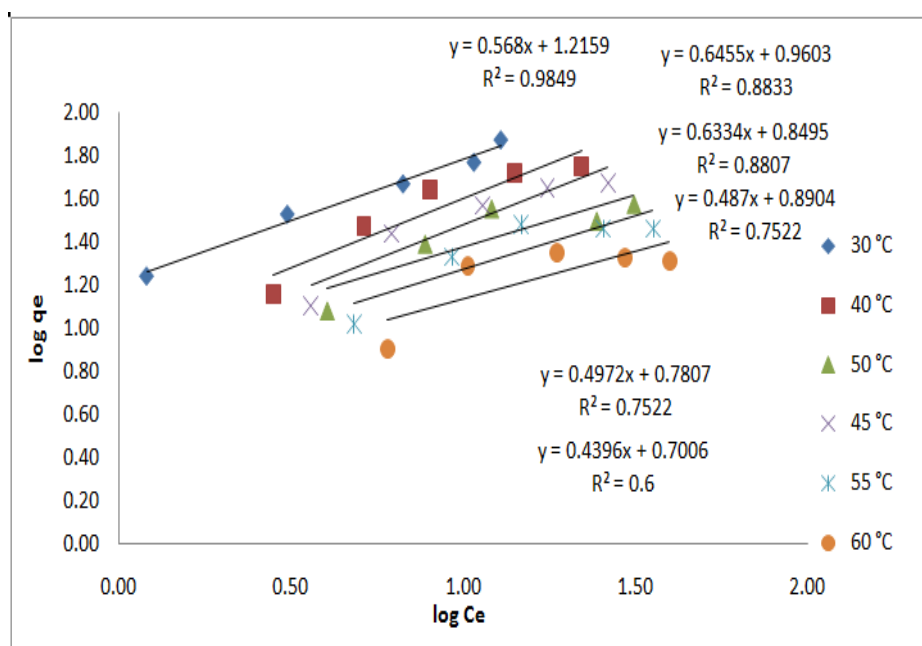


Fig. 9. Freundlich isotherm for the adsorption of methylene blue onto prepared activated carbon

Table 3 Freundlich isotherm parameters

Temperature(°C)	Statistical parameters/constants
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	R^2	n	K_f (mg·L ⁻¹)
30	0.984	1.7605	16.4059
40	0.883	1.5503	9.1201
45	0.880	1.5798	7.0632
50	0.752	2.0534	7.7624
55	0.752	2.0121	6.0256
60	0.6	2.2779	5.0119

The value of R^2 was used to measure goodness of fit of the experimental results obtained on the adsorption isotherm models [30, 26]. The higher values of K_f (16.4059 to 5.0119) indicate that copper pod has greater affinity and higher adsorption capacity for methylene blue molecules. Then values were >1 (in the range of 1.7605 to 2.2779), which indicates the favourable adsorption. The result shows that Freundlich adsorption isotherm is applicable to describe the methylene blue adsorption onto activated carbon.

3.10. Analysis of adsorption kinetics

The study of adsorption kinetics is desirable as it provides information about the mechanism of adsorption or the factors affecting or controlling adsorption rate. Several models can be used to examine the adsorption kinetics process. Kinetic models can be used to analyse the rate of the adsorption and the rate-limiting step. The most widely used kinetic models for the adsorption process are pseudo-first-order, pseudo-second-order and intraparticle diffusion model. In this study, the adsorption kinetics for the adsorption of methylene blue on the activated carbon prepared from copper pod was analysed using the pseudo-first-order and pseudo-second-order models.

3.11. Pseudo-first-order kinetic model

The rate constants k_1 and q_e were determined by plotting $\log (q_e - q_t)$ against t , which is shown in Fig. 10, and the results are shown in Table 4. The calculated q_e values of pseudo-first-order kinetic model do not agree with the experimental q_e values at all the concentrations [31, 25]. The correlation coefficient R^2 values obtained for pseudo-first-order kinetic model were relatively small. The results suggested that the adsorption of methylene blue by activated carbon prepared from copper pod does not follow the pseudo-first-order kinetic model.

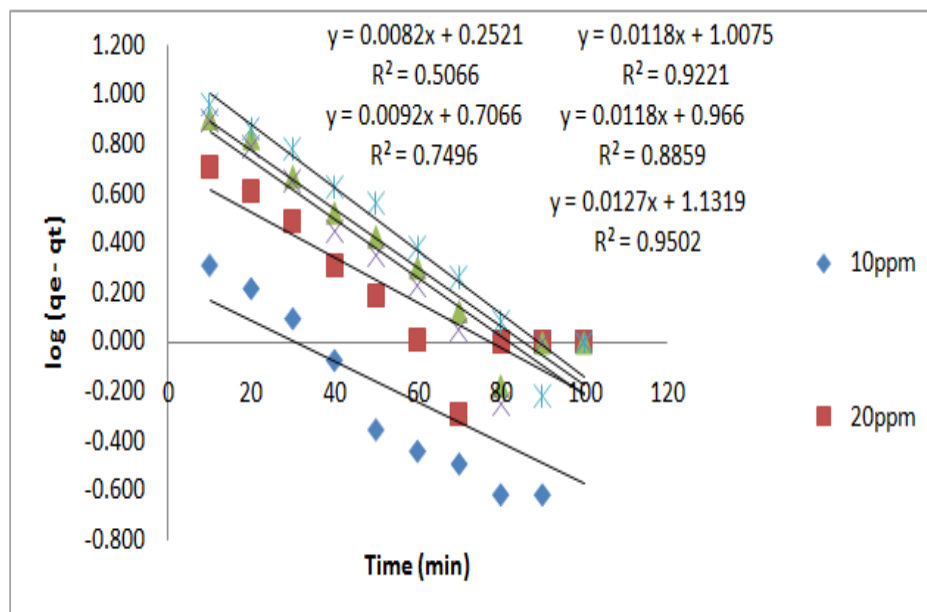


Fig.10. Pseudo-first-order kinetics for the adsorption of methylene blue onto prepared activated carbon at 30°C.

3.12. Pseudo-second-order kinetic model

The rate constants k_1 and q_e were determined from the slopes and intercept of the linear plots of t/q_t versus t , which is shown in Fig. 11. A comparison of the first-order reaction rate constant k_1 , the second-order reaction rate constant k_2 and correlation coefficient R^2 values estimated from the pseudo-first-order and pseudo-second-order equations is shown in Table 4. The correlation coefficient R^2 value obtained for pseudo-second-order kinetic model is higher than that of the pseudo-first-order kinetic model [31, 32]. The data obtained were

well suitable to the pseudo-second-order rate equation with correlation coefficient R^2 values. This result indicated a good agreement between the experimental and the calculated q_e values. On the basis of the high correlation coefficient R^2 values, it can be concluded that the adsorption of methylene blue on activated carbon prepared from copper pod is well-suitable to the pseudo-second-order kinetic model compared with the pseudo-first-order model. It also suggests that the chemisorption process could be the rate-limiting step in the adsorption process.

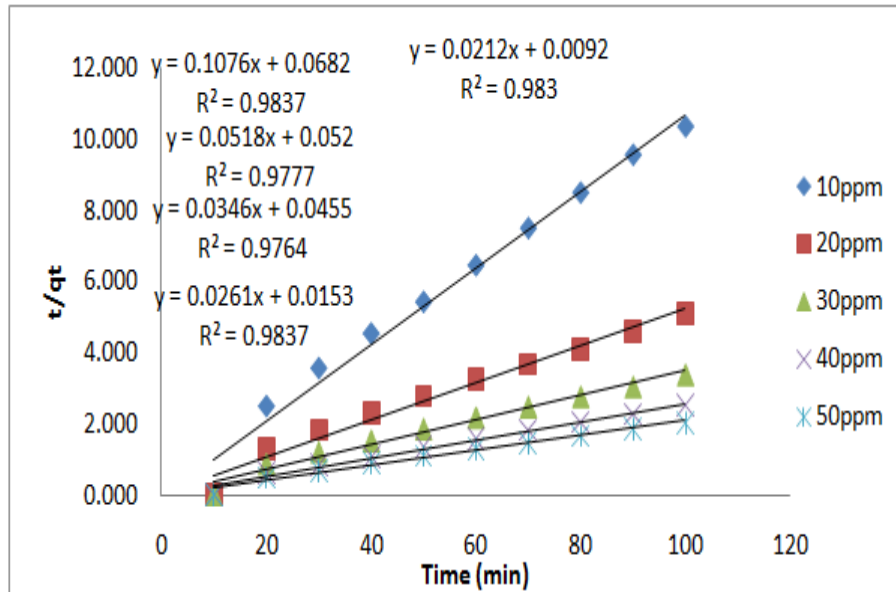


Fig.11. Pseudo-second-order kinetics for the adsorption of methylene blue onto prepared activated carbon at 30°C.

Table 4 ePseudo-first-order and pseudo-second-order kinetic parameters for different initial dye concentrations

Initial concentrations (ppm)	Pseudo-first-order kinetic model				Pseudo-second-order kinetic model			
	q_{eexp} (ppm)	q_{ecal} (ppm)	k_1 (ppm)	R^2	q_{eexp} (ppm)	q_{ecal} (ppm)	k_2 (ppm)	R^2
10	9.64	1.78	0.0184	0.506	9.64	9.34	0.1683	0.983
20	19.49	5.08	0.0207	0.749	19.49	19.60	0.0500	0.977
30	29.33	10.16	0.0253	0.922	29.33	29.41	0.0256	0.976
40	39.44	9.24	0.0253	0.885	39.44	38.46	0.0450	0.983
50	48.78	13.52	0.0276	0.950	48.78	47.61	0.0490	0.983

3.13. Intraparticle diffusion model

A plot of q_t versus $t^{1/2}$ gave a straight line (Fig.12) and the k_{diff} and C values could be determined from the slope. Values are presented in Table 5. C values indicated that the information about the thickness of the boundary layer and the external mass transfer increased as the intercept increased. The linear plots at each concentration did not pass through the origin. The slight deviation of straight lines from the origin indicated that the difference in the rate of mass transfer in the initial and final stages of adsorption and the pore diffusion was only the rate-limiting step [33, 34]. The higher value of correlation coefficient clearly indicated that the pore diffusion played a significant role in the adsorption of methylene blue onto the activated carbon prepared from copper pod. This result also indicated that the intraparticle diffusion process was supposed to be the rate-limiting step.

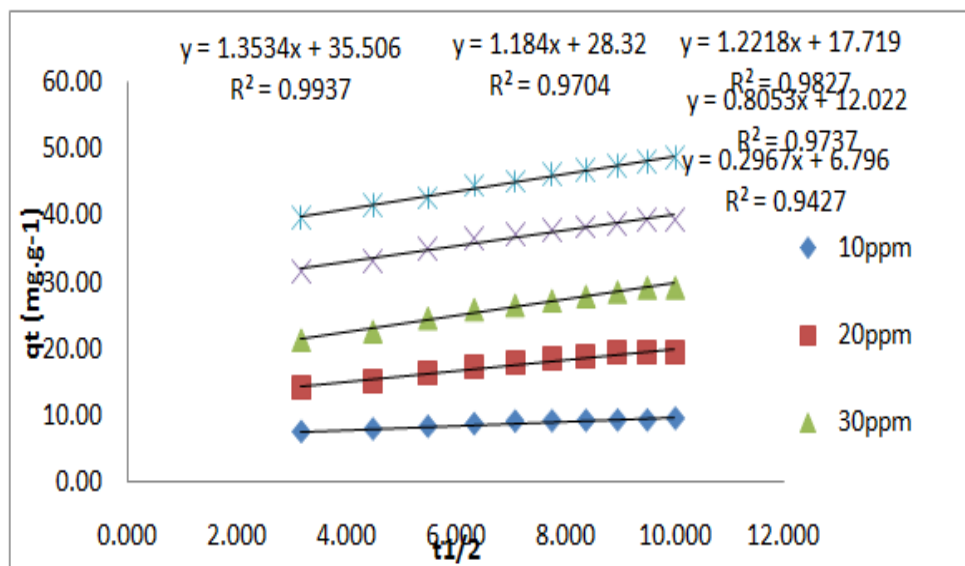


Fig. 12. Intraparticle diffusion model for the adsorption of methylene blue onto prepared activated carbon at 30°C

Table 5 Intraparticle diffusion model

Initial concentration (ppm)	Intraparticle diffusion model		
	C	K_d ($\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-1}$)	R^2
10	35.50	1.353	0.993
20	28.32	1.184	0.970
30	17.71	1.221	0.982
40	12.02	0.805	0.973
50	6.796	0.296	0.942

3.14. Physicochemical characterisation of activated carbon

Physical characteristics of activated carbon prepared from copper pod are given in Table 6. The physicochemical properties such as pH, conductivity, moisture content, ash content, bulk density, specific gravity, water-soluble matter, acid-soluble matter, iodine number ($\text{mg}\cdot\text{g}^{-1}$), surface area ($\text{m}^2\cdot\text{g}^{-1}$) and phenol adsorption capacity were determined for the activated carbon.

Table 6 Physicochemical parameters of activated carbon prepared from copper pod

Parameters	Values
pH	6.95
Conductivity($\text{mS}\cdot\text{cm}^{-1}$)	0.17
Moisture content (%)	3.22
Ash content (%)	18.1
Bulk density ($\text{g}\cdot\text{L}^{-1}$)	0.61
Specific gravity	0.79
Surface area ($\text{m}^2\cdot\text{g}^{-1}$)	886
Iodine number($\text{mg}\cdot\text{g}^{-1}$)	851
Zero point charge in pH units	7.3
Water-soluble matter (%)	0.81
Acid-soluble matter (%)	0.50
Decolorizing power ($\text{mg}\cdot\text{g}^{-1}$)	30
Phenol adsorption capacity($\text{mg}\cdot\text{g}^{-1}$)	5.83

IV. CONCLUSION

The adsorption process is dependent on carbon dosages, contact time, pH and temperature. The adsorption parameters for Freundlich and Langmuir isotherms were determined. The result was very well described by Langmuir and Freundlich isotherm models. The adsorption processes for methylene blue were found to follow pseudo-second-order kinetics. The same results followed for the pseudo-second-order kinetic model and the intraparticle diffusion model. From XRD, SEM studies, it is clear that copper pods act as a valuable adsorbent for the removal of dye from an aqueous solution. Finally, it is concluded that the adsorbent prepared from copper pod could be a good alternative for the removal of methylene blue from an aqueous solution very effectively and economically.

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