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RESEARCH ARTICLE

STUDIES ON ADSORPTION EQUILIBRIUM, ISOTHERMS AND KINETICS OF CONGO RED DYE ONTO SENNA OCCIDENTALIS SEEDS ACTIVATED CARBON

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ABSTRACT

The adsorption potentials of Senna occidentalis Seeds (SoS) Activated Carbon (AC) on the removal of Congo red dye from aqueous solutions has been studied by employing equilibrium adsorption technique, adsorption isotherm and kinetics models. The equilibrium adsorption parameters varied were initial dye concentration, carbon dosage, contact time, pH and temperature. Langmuir, Freundlich and Temkin were chosen as the isotherm models while pseudo first-order, pseudo second-order and intra-particle diffusion were chosen as the kinetics models. The correlation coefficient (R^2) found at 0.9962 and dimensionless separation factor (R_L) at 0.0099 has confirmed that the best fitted model that described the adsorption process of CR dye is the Langmuir adsorption isotherm. Similarly, kinetics of the adsorption processes have been studied and found to be pseudo second-order kinetics. Based on this research findings, Senna occidentalis Seeds Activated Carbon can be said to have potentials for dye removal from effluents of industries, such as; textile, leather, paper, rubber, cosmetics etc.

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INTRODUCTION

Dyes are widely used in industries such as textiles, paper, tanneries, plastics, rubber, cosmetics, etc., to colour their products. Due to their chemical structures, dyes are resistant to fading on exposure to light, water and many chemicals as such, are difficult to be decolourised once released into the aquatic environment (El-Zomrawy and Thabet, 2016). Many dyes are often left as major waste in the industries that used colorants and left to reach our water bodies untreated or poorly-treated (Raghuvanshi *et al*., 2008). Whereas, Shiekh *et al*., (2009) has reported that many of the dyes are hazardous to aquatic animals as they directly affect their life or indirectly affect their foods. The way in which dyes affect lives is in two ways; (1) complex chemical structures and resonating nature of some dyes affect their organs directly, and; (2) some dyes' intense colour do reduces sunlight transmission into water thereby preventing aquatic plants to effectively carry out photo synthesis and thus disrupt aquatic ecosystem. Mahmoodi *et al*., (2013) reported that apart from their effects to aquatic lives, many dyes are also toxic to humans. The removal of dyes from industrial waste before they are discharged into the water bodies is therefore very important from health and hygiene point of view and for environmental protection (Subhrajyoti, 2010). Several methods exist for treating wastewaters consisting majorly physical, chemical and biological processes among which include; adsorption (Mahmoodi *et al*., 2013; Santhi and Smitha 2015), membrane filtration (Mahmoud *et al*., 2007; Yang, 2013), nanofiltration (Oatley-Radcliffe *et al*., 2017), electro coagulation (Nandi and Patel, 2013), coagulation (SDWF, 2015) precipitation (Gupta *et al*., 2012), electrochemical oxidation (Jain *et al*., 2004; Rajeev *et al*., 2004 and Riera-Torres *et al*., 2010), advanced chemical oxidation (Wang and Xu, 2012), Ozonation (Colindres *et al*., 2010; Wijannarong *et al*., 2013), Photo-catalysis (Giwa *et al*., 2012; Ukanah *et al*., 2015), or photo electro catalysis (El-Zomrawy and Thabet, 2016), biodegradation (Ali *et al*., 2009; Ali 2010), etc. But, adsorption technique has been considered as one of the most cost-effective method (Zang *et al*., 2013).

Aim and objectives: Aim of the present study is to evaluate the adsorption potentials of Senna occidentalis Seeds (SoS) activated carbon (AC) for the removal of Congo red dye from an aqueous solution. The objectives are to carry out; (1) Equilibrium studies, (2) Adsorption isotherm studies and, (3) Kinetics study of the adsorption process.

Justification: Despite the challenges associated to the several methods of removing colours, adsorption process has been found to be reasonably efficient and economical process for dyes and pigments removal (Wong *et al.,* 2003). It has also been found to be superior to other techniques for wastewater

treatment in the light of its initial costs, simplicity of design, ease of operation and insensitivity to toxic substances (Garg*et al.,* 2004). Adsorption using activated carbon is rapidly becoming a prominent method of treating aqueous effluents and has been used in industrial processes for variety of separation and purification process, nowadays. Meanwhile, Senna Occidentalis Seeds Activated Carbon (SoS AC) has been developed and reported to have good adsorption potentials although it is yet to be evaluated using any dye effluents (Gulumbe *et al*., 2019).

MATERIALS AND METHODS

Preparation of adsorbent: The adsorbent used in this study is activated carbon prepared from *Sena occidentalis* seeds and was adopted from the research conducted by Gulumbe *et al*., (2019). It is intended to evaluate and confirm the findings of that research. Similarly, the preparatory methods were reported in the already mentioned work (Gulumbe *et al*., 2019).

Preparation of adsorbate: Congo Red (CR) dye is used in these studies as the adsorbate. CR is a secondary disazo dye, a sodium salt of benzidinediazo-bis- 1-naphthylamine-4-sulfonic acid (molecular formula: $C_{32}H_{22}N_6Na_2O_6S_2$; molecular weight: 696.66 g/mol and λmax = 500 nm). The stock solution of CR was prepared by dissolving 1g of its powdered dye into 1000 ml of double distilled water obtained from Chemical Engineering Laboratory, A.B.U, Zaria. The dye purchased from Cardinal Scientific Supplies, Zaria, Nigeria, was used as received and successive dilutions were made to obtain the working solution of desired concentrations.

Batch adsorption studies: Adsorption experiment was conducted by adding a mass (in g) of activated carbon into 100 ml dye solution of a known concentration (in mg/L) in a 250 ml beaker at a ambient temperature (of around 30 ^{0}C), and the mixture was stirred on a magnetic stirrer at 100 rpm. The samples were withdrawn from stirring setup at set time intervals, and the adsorbent was separated from the solution by the help of a micropipette, and then allowed to settle for 10 minutes. 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 at the maximum wavelength of 500 nm. The above described method was repeated in a batch adsorption process, it was carried out to investigate the effects of variables such as; initial dye concentration (10–60) mg/L, contact time (20–180) minutes, pH (3–11) adjusted using 0.1M NaOH and 0.1M HCl solution and a pH meter. The effects of temperature and carbon dosage were also evaluated at 303, 313 and 323 K, and between the ranges of 0.5 g – 2.5 g respectively.

The amount of CR dye adsorbed at equilibrium (q_e) and time, t (q_t) – all in mg/g, were calculated using the eq. 1 and 2 respectively, as well as the percentage dye removal (%) using the eq. 3 (Subhrajyoti, 2010):

$$qe = \frac{(C_\circ - Ce) \times V}{M} \quad (1)$$

$$qt = \frac{(C_\circ - Ct) \times V}{M} \quad (2)$$

$$R\ (\%) = \frac{(C_\circ - Ce)}{C_\circ} \times 100 \quad (3)$$

where; C_0 and C_e are the initial and equilibrium concentrations of the dye in solution (mg/L), C_t is the equilibrium dye concentration at time, t (mg/L), V is the volume of the solution used (L) and M is the mass of the adsorbent (g). In each case, the parameter tested was plotted against the percentage dye removal (%) using equation (3).

RESULTS AND DISCUSSION

Batch Adsorption Studies

Effect of initial dye concentration: The effect of initial CR dye concentration on the percentage CR dye removal was obtained by varying the concentrations from 10mg/L to 60mg/L, leaving other parameters constant. The result is presented in the Fig. 2.

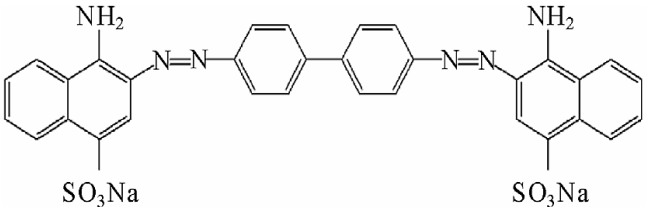

Fig. 1. Chemical Structure of Congo Red Dye

Effect of contact time: The effect of contact time on the percentage CR dye removal has been studied and presented in Fig. 3. It is clear that the extent of CR dye removal was rapid in the initial stages and becomes slow in later stages till saturation was attained. This primarily might be due to the saturation of the active sites on the SoS AC which do not permit further adsorption to occur. However, this can be explained by the fact that there is very large quantity of sites on the surface of SoS AC at the initial stages, which allowed adsorption took place very easily. But with the passage of time, the active sites got saturated thereby reducing the rate at which adsorption or removal occurred. The saturation can be said to be achieved at least after 140 min.

Effect of activated carbon dose: The result for variation of percentage removal of CR with adsorbent doses of 0.5g, 1g, 1.5g, 2g and 2.5g is presented in Fig. 4. The figure shown that increase in the quantity of SoS AC resulted in a corresponding increase in the percentage CR dye removed. The increase in CR dye removal with simultaneous increase in carbon dose might be due to the increase in surface area which translates to availability to more sites for adsorption of the dye. Meanwhile, 1g of SoS AC has achieved at least 83% CR dye removal from wastewater and hence, was chosen to be the optimum carbon dose under these studies. This finding is in line with similar report that has been made earlier (Akpen*et. al*., 2011).

Effect of pH: The effect of initial pH on the percentage CR dye removal was investigated by varying solution's pH on a range of 3 to 11 and presented in Fig. 5. The highest CR dye removal was notably at pH = 3, then there was no observable difference in the percentage removal at pH 5 – 7 but minimum at around pH value of 11. Thus, the percentage CR dye removal increased with increased in initial pH of the dye solution.

Effect of temperature: The effect of temperature on the percentage removal of CR dye solution has been determined

and the result is shown in Fig. 6. The Result indicated that the adsorption capacity of SoS AC for the removal of CR dye increases with temperature. This might be due to increase in the mobility of the large dye ions with temperature. Several CR dye molecules might have also acquired adequate energy that enabled them move towards the active sites at the surface. Likewise, increase in temperature might have produced a swelling effect within the spongy structure of the activated carbon enabling large dyes to penetrate inside.

Adsorption Isotherms

Langmuir adsorption isotherm: The Langmuir adsorption is based on the view that every adsorption site is identical and energetically equivalent (i.e. thermodynamically, each site can only hold one adsorbate molecule). It was based on the assumption that the eq. 4 and 5are formed (Langmuir, 1916 and Langmuir, 1918):

$$C_t/q_t = 1/(q_m \times b) + (1/q_m) \times C_t \quad (4)$$

$$R_L = 1/(1+bC_0) \quad (5)$$

where; C_t is the equilibrium concentration in solution at time, t (mg/L), C_0 is the highest initial concentration (mg/L), q_e is the amount of dye adsorbed at time, t (mg/g), q_m is the constant related to overall solute adsorptivity (L/g) and R_L is the shape of the adsorption isotherm. A plot of C_tq_t against C_t gives a graph of $1/q_m$ as slope and $1/q_m \times b$ as intercept. The Langmuir constant, separation factor R_L value obtained from Fig. 6 was 0.0099 for CR dye adsorption which indicates that the adsorption was favourable ($0<R_L<1$). The monolayer adsorption (q_m) and correlation coefficient (R^2) were found to be 1.97 mg/g and 0.9962 for CR dye adsorption respectively. These results have indicated that the description of adsorption process by means of Langmuir isotherm model is most favourable and fit.

Freundlich adsorption isotherm: Freundlich isotherm is commonly used to describe the adsorption characteristics for the heterogeneous surface. The plot of ln versus ln yields (1/n) as slope and ln K_f as the intercept. Freundlich isotherm model shownas eq. 6 (Freundlich, 1906; Xunjun, 2015):

$$\text{Lnqt} = \ln K_f + (1/n) \ln C_t \quad (6)$$

The Freundlich constants K_f and n which stands respectively for adsorption capacity and adsorption intensity have indicated the isotherm process to be favourable. It was established in the literature (Ho and Chang, 2001; Okoli *et al*., 2015) that the favourability of the adsorption process is said to be achieved if n value lies in the range of 1 – 10. The adsorption intensity (n) and correlation coefficients (R^2) obtained from Fig. 8 were found to be 8.772 and 0.9569 for CR dye, hence, favourable.

Temkin adsorption isotherm: The Temkin isotherm assumes that the fall in the heat of sorption is linear rather than logarithmic, as implied in the Freundlich equation. The isotherm is derived by assumption that for adsorption at a single site, the appropriate equation is assumed to be eq. 7 (Tempkin and Pyzhev, 1940):

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (7)$$

$$B_1 = RT/b_T \quad (8)$$

The Temkin isotherm constants such as equilibrium binding constant, K_T (L/g) and Temkin heat of sorption, B_1 (J/mol) were respectively obtained from the intercepts and slopes of plot of q_e against ln C_e. While, Temkin isotherm constants, b_T is derived from eq. 8. However, the correlation coefficient (R^2) of Temkin isotherm for CR dye adsorption was found at 0.8420.

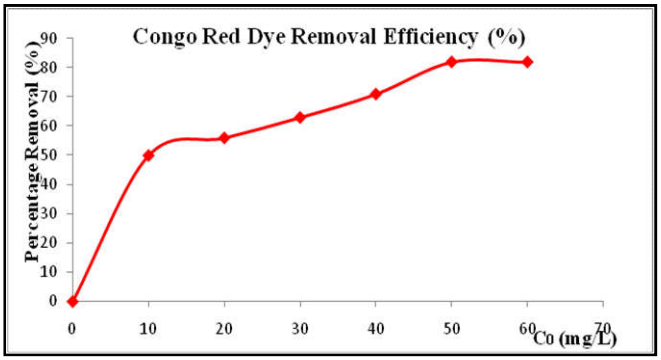

Fig. 2. Effect of Initial Dye Concentration on the Percentage CR Dye Removal onto SoS AC (at AC Dose = 1.5g, Temp. = Ambient Temp., Contact Time = 60min, pH = Dye pH and Speed – 100rpm).

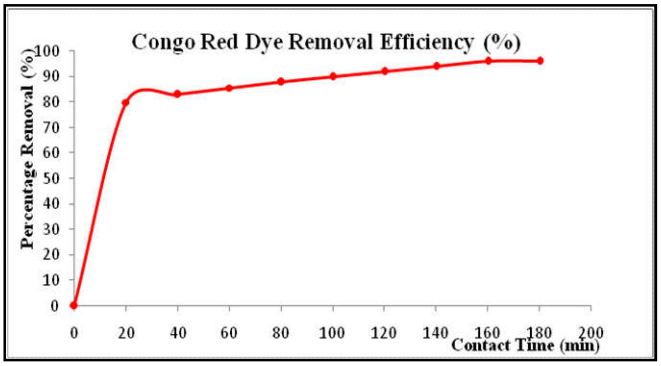

Fig. 3. Effect of Contact Time on the Percentage CR Dye Removal onto SoS AC (at C_0 = 50mg/L, SoS AC Dose = 2g, pH = Dye pH, Temp. = Ambient Temp. and Speed = 100rpm).

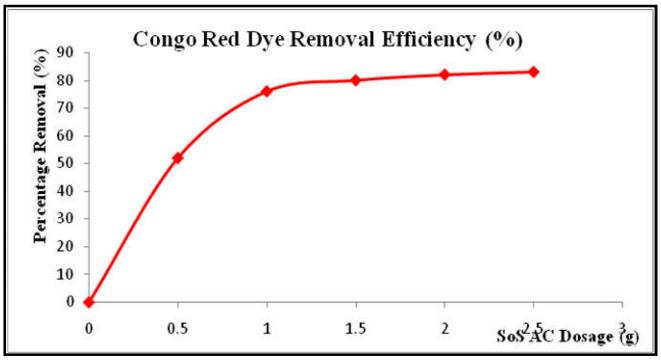

Fig. 4. Effect of Carbon Dosage on the Percentage CR Dye Removal onto SoS AC (at C0 = 50mg/L, pH = Dye pH, Temp. = Ambient Temp., Contact Time = 60min. and Stirring Speed = 100rpm).

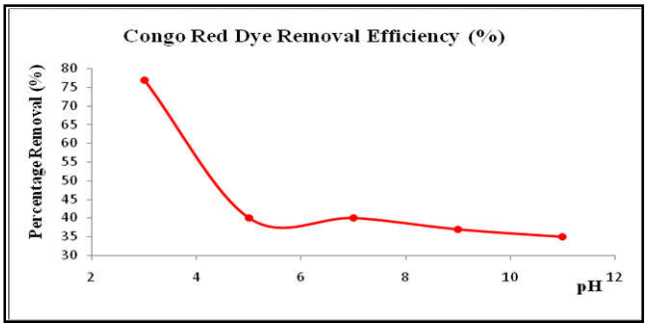

Fig. 5. Effect of Initial pH of Dye Solution on the Percentage of CR Dye Removal onto SoS AC (at C_0 = 30 mg/L, Contact Time = 40 min., Temp = Ambient Temp., SoS AC Dose = 1g/100ml and Stirring Speed = 100rpm).

Kinetics Studies

Pseudo first-order kinetic model: The Pseudo-first order adsorption rate constants, K_1 (min^{-1}) and calculated q_e for CR dye was determined from the slope and intercept of plot of ln ($q_e - q_t$) against t as derived from eq. 9 (Lagergren 1898) and presented in Fig. 10. The results of the q_e and K_1 were evaluated and listed in the Table 3.2. The value of the correlation coefficient (R^2) was determined at 0.9905 for CR dye.

$$\log (q_e - q_t) = \log (q_e) - (k_1 t)/2.303$$

Pseudo second-order kinetic model: The pseudo second order adsorption kinetic rate equation as expressed by (Roshan, 2015) is shown as eq. 10 and its integrated and linearized form is given as eq. 11:

$$dq_t/dt = k_2 (q_e - q_t)^2 \quad (10)$$

$$t/qt = 1/ (k_2 q_e^2) + t/q_e \quad (11)$$

A plot of t/qt versus t should give a straight line if this model is obeyed by the sorption process. From the slope and intercept of the plots, q_e and k_2 are determined, respectively. However, the experimental q_e is expected to tally with the estimated one. Decrease in the values of k_2 suggests increased adsorption. It can be seen that pseudo second-order rate expression in Fig. 11 has value of correlation coefficient (R^2) of 0.9986 for CR dye adsorption and hence, fit better than the former. Similarly, the results of kinetic rate constants of pseudo-second order (K_2) is better than its corresponding kinetic rate constants of the pseudo-first order (K_1) as shown in Table 3.2.

Intra-particle diffusion model: The logarithmic form of the intra-particle diffusion model is given as eq. (12):

$$\log q_t = \log k_{id} + 0.5 \log t \quad (12)$$

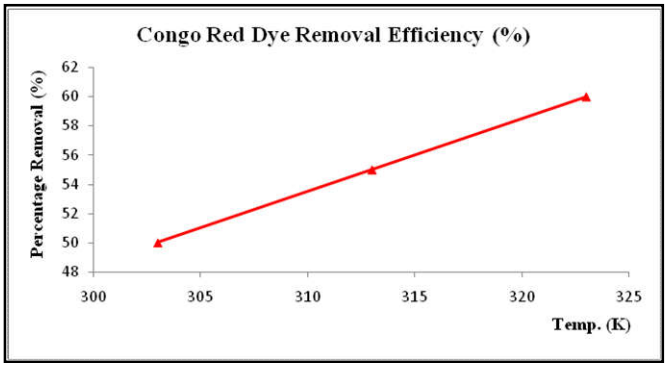

Fig. 6. Effect of Temperature on the Percentage CR Dye Removal onto SoS AC (at C_0 = 20 mg/L, pH = Dye pH, Carbon Dose = 0.5g/100ml, Contact Time = 40 min. and Speed = 100 rpm)

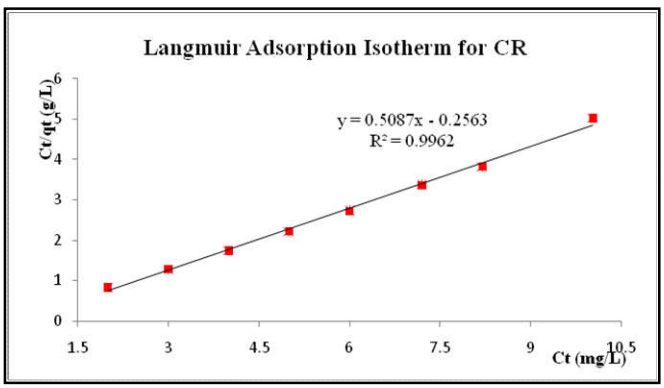

Fig. 7. Langmuir Isotherm Plot for Adsorption of CR Dye onto SoS AC

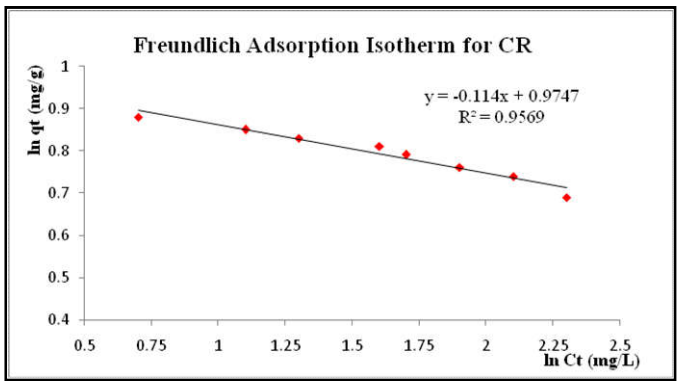

Fig. 8. Freundlich Isotherm Plot for Adsorption of CR Dye onto SoS AC

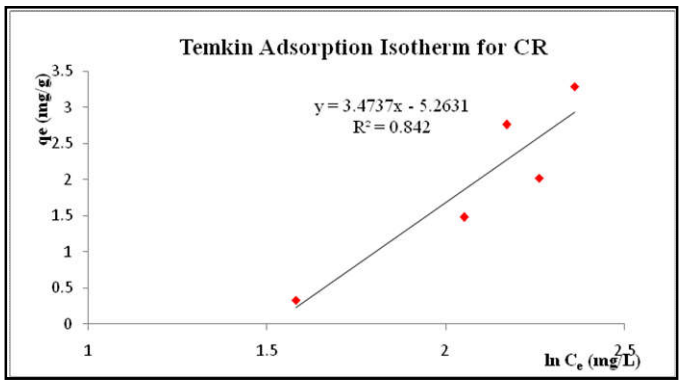

Fig. 9. Temkin Isotherm Plot for Adsorption of CR Dye onto SoS AC

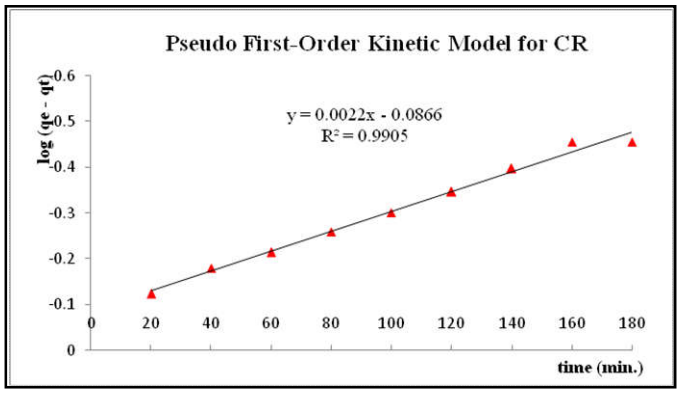

Fig. 10. Pseudo First-order Kinetics Model Plot for Adsorption of CR Dye onto SoS AC

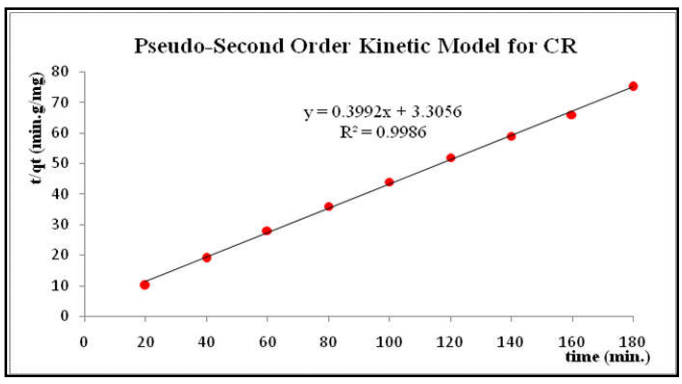

Fig. 11. Pseudo Second-order Kinetics Model Plot for Adsorption of CR Dye onto SoS AC

The plot of log q_t against 0.5 log t should yield a straight line with a positive intercept for intra-particle diffusion controlled adsorption process. k_{id} is determined from the intercept of the plot. Higher values of K_{id} illustrate an enhancement in the rate

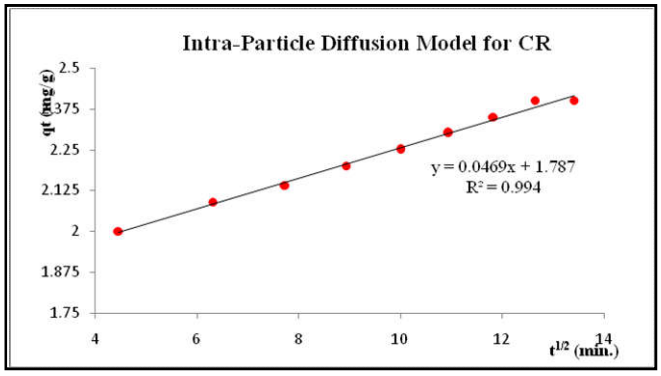

Fig. 12. Intra-particle Diffusion Kinetics Model Plot for Adsorption of CR Dye onto SoS AC

Table 1. Isotherm Constants for the Adsorption of CR Dye onto SoS AC at 303K

Models	Parameters	Congo Red Dye
Langmuir	K_L (L/mg) Q_m (mg/g) R_L R^2	1.89 2.0 0.01 0.9962
Freundlich	K_f (L/g) 1/n N R^2	2.65 0.114 8.77 0.9569
Temkin	K_T (L/mg) b_T (J/mg) B_1 (J/mol) R^2	4.5 713.23 3.47 0.8420

Table 2. Constants from Kinetic Studies of CR Dye Adsorption onto SoS AC at 303K

Models	Parameter	Congo Red Dye
Pseudo-first Order	K_1 (min^{-1}) qe (mg/g) R^2	2.20×10^{-3} 0.09 0.9905
Pseudo-second Order	K_2 (g/mg/min) q_e (mg/g) R^2	3.31×10^{0} 0.40 0.9986
Intra-particle Diffusion	K_{id} (mg/min.g) C_{id} (mg/g) R^2	0.05 1.79 0.9940

of adsorption. The intra-particle diffusion model was tested to find out whether the intra-particle diffusion step has any influence over the rate-controlling step of CR dye adsorption onto SoS AC. The diffusion constants, K_{id} and C_{id} were determined from the slope and intercept of the plot of q_t against $t^{0.5}$ as presented in Fig. 12. However, the results of K_{id}, C_{id} and R^2 were evaluated and listed in Table 2. The value of the correlation coefficient (R^2) obtained was 0.9940 for CR dye, which its magnitude suggests the presence of intra-particle diffusion as the rate determining step. While, the rate limiting step is the lowest step of the reaction which may be the intra-particle diffusion or the boundary layer of solute on solid surface from the bulk of the solution in a batch process. In short, when the linear lines of the model plots deviate from passing through the origin (due to the difference in mass transfer rate from the initial to the final stages of adsorption), that indicates that intra-particle transport is not the 'one and only' rate limiting step, instead other kinetic models may simultaneously control the rate of adsorption.

Conclusion

Removal of CR dye from aqueous solutions by adsorption technique using SoS AC has been the primary focus of this research and the following conclusions were reached:

- It is obvious from this research that percentage CR dye removal increased with increased in all the tested variables except in pH. The results have shown; 82% CR dye removal during study of the effect of initial dye concentration.
- Optimum contact time for equilibrium achieved was found at around 160 min. It is basically due to saturation of the active sites which did not allow further adsorption. While, the optimum carbon dose for the dye removal was found to be 1.5g/100 ml, due to the active sites saturation. Other optimum conditions include; temperature at 50 and pH at 3.
- Langmuir, Freundlich and Temkin adsorption isotherm were all successfully tested and for the purpose of comparison, Langmuir isotherm was found to be the most suitable model that best described the adsorption process. Similarly, of all the three kinetic models so far tested, the pseudo-second order kinetic model was the best fit and able to better described the adsorption kinetics process of CR dye onto SoS AC.

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