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RESEARCH ARTICLE

USE OF CHARCOAL DERIVED FROM BAMBOO SEED FOR ADSORPTION OF DYES

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ABSTRACT

In this work, adsorption of Rhodamine 6G, Carmine, Fast Green FCF,Safranine O from aqueous solution onto activated charcoal were prepared from Bamboo seed seed by physicochemistry activation with potassium hydroxide (KOH) as the activated agent at 700°C for 1hour and particle size 600µm analyzed results by U.V-Visible technique .The influence of variables parameters including PH, amount of adsorbent, sieve size of adsorbent, temperature and contact time on dyes removal were studied. The isotherm data was analyzed by Langmuir, Freundlich, Tempkin,Harkins-Jura and Dubinin-Radushkevich. Calculation of various thermodynamic parameters such as enthalpy (ΔH), entropy (ΔS), Gibb's free energy changed (ΔG), activation energy (Ea), sticking probability (S*). The positive values of (ΔG) indicate that the dyes adsorption process is unspontaneous in nature and the negative values of (ΔH) shows the exothermic nature of the process. The adsorption kinetics shows that, pseudo first order and pseudo second order rate equation, pseudo second order was fitted better than pseudo first order.

INTRODUCTION

Activated carbon can be used for removal of Rhodamine 6G, Carmine, Fast Green FCF, Safranine O in effluent or coloured substances from gas or liquid streams by the mechanism of adsorption. Adsorption can be classically defines as absorption on the surface of the material due to capillary condensation inside the multitude of pores, active sites available. In its effort to promote value added Bamboo seed based products, the national mission on Bamboo seed applications lunched an investigation to study the conversion of Iraqi Bamboo seed species to activated carbon in association with Iraqi institute of technology-Bamboo seed. Bamboo seed is the general name used for a number of perennial, woody-stemmed grasses. Native cane (Arundinaria gigantean), which is commonly referred to as river cane, grows naturally in Kentucky and throughout much of the southeast. It is one of three Bamboo seed species native to north America[1]. Synthetic dyes are one of the main pollutant groups of water and wastewater. Dye contamination in wastewater causes problems in several ways: the presence of dyes in water, even in very low quantities, is highly visible and undesirable;color[2]: most dyes are considered to be non-oxidizable substances by conventional biological and physical treatment because of their complex structure and large molecular size. The adsorption process provides an attractive alternative treatment, especially if the adsorbent is in expensive and readily available[3].Colour from aqeous medium is usually removal with tertiary treatment such as coagulation, floatation, oxidation hyper filtration and adsorption [4]. The provision of safe water to the people is an urgent development priority of the world. The continual expansion and increasing of Al-Najaf centers in developing nations has resulted in pollution of water sources [5]. The dying effluent discharged from textile industries is one of the largest contributors to textile effluent and is comprised mainly of residual dyes and auxiliary chemicals. Dying effluent has a serious environmental impact because disposal of this effluent into the receiving water body damages aquatic biota or humans by mutagenic and carcinogenic effects[6]. The presence of very low concentrations of these effluents are highly visible and undesirable and potentially inhibiting photosynthesis. The main pollution source of colored effluents comes from textiles, leather. Printing, laundry, rubber, painting, etc, processes[7] untreated disposal of this colored water into receiving water body causes damage to aquatic life and also severe damage to aquatic life and also severe damage to the human health [8-10].

MATERIAL AND METHODS

Material

Charcoal Derived from Bamboo seed (CDB) was used in this study. The samples were washed several times and dried in an oven at 100°C and was ground into fine powder form before being used. Fast Green FCF, Rhodamine 6G, Carmine, Safranine O supplied by Merck, BDH respectively were used. The dyes having molecular formula ($C_{37}H_{34}N_2Na_2O_{10}S_3$, $C_{28}H_{31}N_2\ O_3\ Cl$, $C_{22}H_{20}O_{13}$ and $C_{20}H_{19}N_4Cl$) with molecular weight (808.86, 479.02, 492.39 and 350.85).

Preparation of dyes solution

The dyes stock solution was prepared by dissolving accurately weighted dyes in distilled water to the concentrations of 50mg/L. The experimental solutions were obtained by diluting the dye stock solution in accurate proportions to different initial concentrations.

Preparation of Charcoal Derived from Bamboo seed (CDB)

Bamboo seed used for preparation of Charcoal Derived was procured locally, washed, dried, crushed to desired mesh size 600µm. The raw material was then carbonized at 700°C under nitrogen atmosphere for 1 hour. A certain amount of produced char then was soaked with potassium hydroxide (KOH) at impregnation ratio of 1:1. The mixture was dehydrated in an oven overnight at 105°C; then pyrolysed in a stainless steel vertical tubular reactor placed in a tube furnace under high purity nitrogen 99.99% flow of $150cm^3min^{-1}$ to a final temperature 0f 850°C and activated for 2hour.

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Once the final temperature was reached, the gas flow was switched to carbon dioxide and activated was continued for 2hour. The carbon product was then cooled to room temperature under nitrogen flow and washed with deionized water to remove remaining chemical .Subsequently the sample was transferred to a beaker containing a 250ml solution of hydrochloric acid, stirred for 1hour, and then washed with hot deionixed water until the PH of the washing solution reached 6-7 [11]

SORPTION STUDIES

Analysis of dyes

The concentration of Fast Green FCF, Rhodamine 6G, Carmine, Safranine O in the supernatant solution after and before adsorption was determined using UV-Visible spectrophotometer at (527,637,535 and 525 nm). It was found that the supernatant from the charcoal didn't exhibit any absorbance at this wavelength and also that the calibration curve was very reproducible and linear over the concentration rang used in the work (2-20ppm) Fig.1.

Table 1. Break through capacity of dyes

Dyes	Fast Green FCF	Rhodamine 6G	Carmine	Safranine O
	Co=20ppm	Co=20ppm	Co=20ppm	Co=20ppm
volume	Ce/Co	Ce/Co	Ce/Co	Ce/Co
6	0.144	0.233	0.465	0.622
12	0.326	0.481	0.601	0.736
16	0.577	0.732	0.811	0.945

Batch adsorption experiments

Sorption experiments were carried out in a rotary shaker at 200rpm and 30±2°C using 50ml shaking flasks containing 20ml different concentrations and initial PH values of dyes solutions. The initial PH values of the solution were previously adjusted with dilute HCl or NaOH using PH meter. Different doses of charcoal derived from Bamboo seed were added to each flask and then the flasks were sealed up to prevent change of volume of the solution during the experiments. After shaking the flasks for predetermined time intervals, the samples were withdrawn from the flasks and the dyes solution was separated from the sorbent by centrifugation. The amount of dyes uptake by charcoal derived from Bamboo seed were calculated using the mass balance equation[13].

$$Qe = \frac{Vsol(Co - Ce)}{m}$$

Co is the initial concentration (mg/L)
Ce is the equilibrium concentration (mg/L)
V is the total volume of BSA (L)
M is the weight of charcoal derived (gm)
Qe is the amount of Adsorption (mg/g)

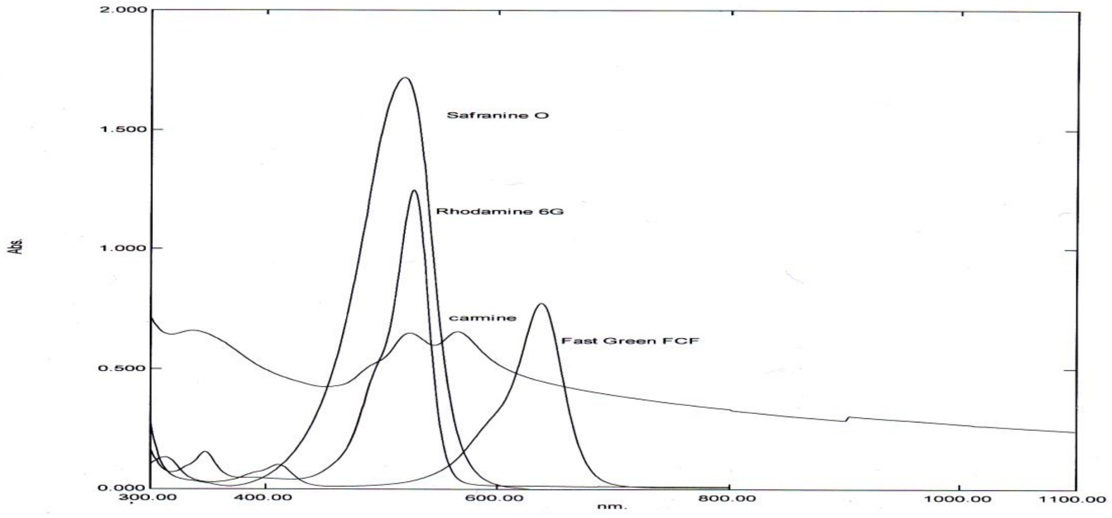

Fig.1. The wavelength for adsorption dyes on CDB at 30°C

Effect of adsorbent dose

The effect of sorbent dose on the equilibrium uptake of dyes was investigated with sorbent masses of 0.01,0.05,0.1,0.15 and 0.2 g. The experiments were performed by adding the known weights of charcoal derived from Bamboo seed to five conical flask 50 ml containing 20ml of 0.01g solution at PH=7. The flasks were shaken at 200rpm and 30°C for optimum contact time and equilibrium concentration of the remaining dye was determined spectrophotometrically.

Effect of PH

The influence of PH for the adsorption of dyes were observed .For this purpose 13 experiments at different PH for each dyes were accomplished 20ml solution of dyes carrying variable PH(2-14) was added in different solutions HCl, NaOH with optimum of weight surface in experimental.

Break through capacity

20 ml of feed solution containing 20ppm of dyes was allowed to pass though the conical flasks containing 0.2g of charcoal derived from Bamboo seed .The process was continued till the amount of dyes was same in feed and effluent. Break though capacity was calculated using the ratio Ce/Co where Ce is concentration of the dyes in the effluent and Co in the concentration of dye in the feed [12] Table 1.

RESULTS AND DISCUSSIONS

Effect of particle size of CDB

The effect of particle size on the quantity of dye adsorbed is given in the Fig.2. The effect of particle size on the quantity of dye adsorbed was studied only by varying the particle size of CDB as (53,212,500,600μm).Effect of particle size of adsorbent on the adsorption was studied at 30±0.5°C, 0.2 g of CDB and dyes concentration of 20mg/l. Adsorption was found to decrease with the increase in the particle sizes because of the decrease in surface area of the adsorbent and accessibility of the adsorbent pores towards the dyes[14].

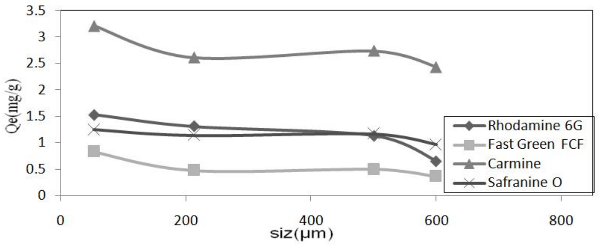

Fig.2. Effect of particle size for adsorption of dyes at 20mg/l and 30°C

Effect of contact time on dyes removal

Equilibrium time is one of the most important parameters in the design of economical wastewater treatment system. The adsorption of dyes on CDB was studied as a function of contact time in order to determine the necessary adsorption equilibrium time. Rapid uptake and quick establishment of equilibrium time imply the efficiency of particular adsorbent in terms of usage in wastewater treatment. Fig. 3 shows the effects of contact time on adsorption of dyes by CDB and it was observed that the adsorption rate is rapid at the initial stages and then gradually increase with the progress of adsorption until the equilibrium is reached [6]. The rapid adsorption at the initial contact time can be attributed to the availability of the positively charged surface of CDB and the slow rate of dyes adsorption is probably due to the slow pore diffusion of solute dye into the bulk of the adsorbent[15]

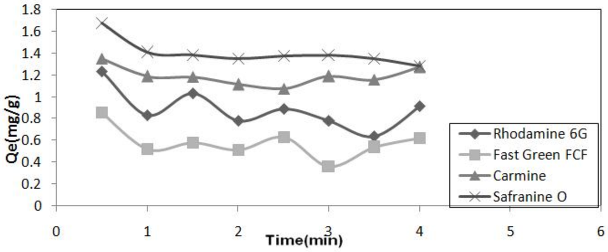

Fig.3. Adsorption kinetics of dyes on CDB at 30°C, 20mgL-1

Effect of PH on dyes adsorption

The wastewater from textile industries usually has a wide range of PH values. Thus PH of the system plays an important role in the textile wastes treatment. The value of PH affects both aqueous chemistry and surface binding sites of the adsorbents. The effect of initial PH on the adsorption of dyes was studies in the range of 2 of 14 at 30°C at initial dyes concentration of 20mg/l, adsorbent dose of 0.2g and contact time of 30min and respective results are present in Fig.4. As it can be seen, the maximum uptake of the Fast Green FCF is obtained at PH of 2, Carmine at PH of 3, Safranine O at PH of 4 and Rhodamine 6G at PH of 7. It was observed, that the PH significantly affects the extent of adsorption of dyes over the adsorbent and a decrease in the adsorption efficiency further this value was observed. The increased competition between H^+ and dyes for available adsorption sites leads to a lower removal efficiency because a large number of sites on CDB may because positively charged at very low PH[16-17]

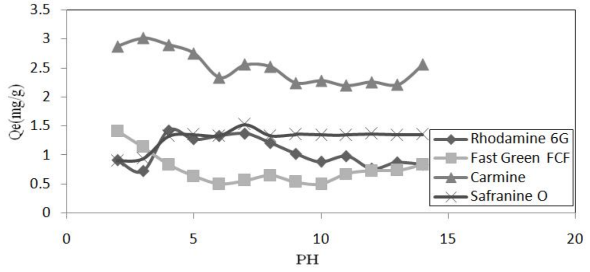

Fig.4. The effect of PH on the adsorption of dyes solutions by CDB at20mg/l and 30°C

Effect of adsorbent dose at constant dyes concentration

The effect of sorbent dose on the equilibrium uptake of dyes was investigated with sorbent masses of 0.01,0.05,0.1,0.15 and 0.2g. The experiments were performed by adding the known weight of CDB to five 50ml conical flask containing 20 ml of 0.01mmol/dm^3 solution at optimum PH. The flasks were shaken at 200rmp at 30°C for 0.5h and the equilibrium concentration of the remaining dye was determined spectrophotometrically. It was observed that highest amount of dyes removal attained for adsorbent mass of at least adsorbent[18], This may be due to the rapid increase in surface and number of available active sites for the adsorption of dyes or due to conglomeration of CDB at higher doses[19]Fig.5.

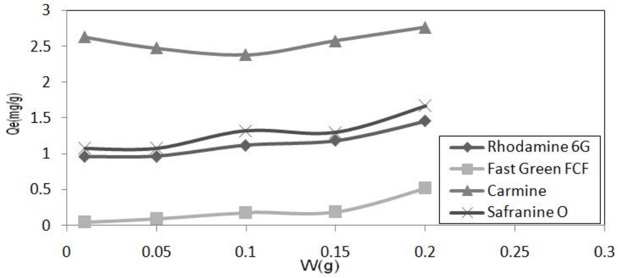

Fig.5. Effect of weight adsorbent for dyes on CDB at 30°C,20mgL^{-1}

Effect of Temperature and Thermodynamic study

For both the adsorption all the adsorption studies were carried out 30 to 50°C temperature and Fig.6. clearly reveal that in each case adsorption decrease with the increase in temperature. Results show that the adsorption of dyes decrease with increase in temperature that indicates the process is exothermic and can be explained by the exothermic unspontaneity of the adsorption process. This decrease in sorption capacity with temperature is due to the enhancement of the desorption step in the sorption mechanism indicating that the process is exothermic [20]. It is known that decreasing sorption capacity with increase temperature is mainly due to the weaking of sorption forces between the active site on the CDB and dyes species, and also between adjacent dye molecules on the sorbed phase. For a conventional mechanism of physisorption system, increase in temperature usually increase the rate of approach to equilibrium, but decrease the equilibrium capacity [21]. Thermodynamic parameters such as change in Gibbs free energy (ΔG), charge in enthalpy (ΔH), and change in entropy (ΔS) of dyes adsorbed by the CDB, are calculated by equation [22-23]

$$\ln K_d=\ln\left(\frac{qe(t)}{Ce(t)}\right)=\frac{\Delta S}{R}-\frac{\Delta H}{RT}$$

$$\Delta G=\Delta H-T\Delta S$$

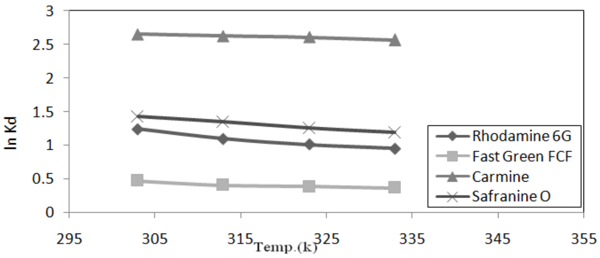

Fig.6. Effect of temperature of dyes adsorption by CDB at 20mgl^{-1} and 30°C

Where Kd is the distribution coefficient of the adsorbate, qe(t)(mg/g) is the dyes adsorption capacity of the CDB at time t, Ce(mg/l) is the dyes equilibrium concentration at time t ,and R(8.314 J/mol.K) is the ideal gas constant. The values of ΔS and ΔH are calculated from the slopes and intercepts of the linear plots of log Kd versus 1/T Fig 7,

ΔG is obtained using Gibb's equation the thermodynamic parameters are listed in Table 2. The negative ΔH value confirms that the adsorption process is exothermic for dyes, which is an indication of the existence of a strong interaction between CDB and dyes. The positive value of ΔG confirmed the unspontaneous nature of adsorption process. The positive of ΔS indicates that the degrees of freedom increased at the solid –liquid interface during the adsorption of dyes onto CDB and reflected the affinity of CDB toward dyes in aqueous solutions and may suggest some structural changes in adsorbents[24]. The values of activation energy (Ea) and sticking probability (S*) were estimated from the experimental data, they were calculated using modified Arrhenius type equation relation to surface coverage (θ) as follows[25]

$$Log\ S^* = Log\ 1 - \theta - \frac{Ea}{2.303\ RT}$$

The sticking probability S*, is a function of the adsorbate, adsorbent system under investigation, its value lies in the range $0 < S^* < 1$ and is depended on the temperature of the system. The parameters S* indicates the measure of the potential of an adsorbate to remain on the adsorbent indefinite. The surface coverage θ can be calculated from the following equation:

$$\theta = 1 - \frac{Ce}{Co}$$

The activation energy and sticking probability were estimated from a plot of log (1- θ) vs. 1/T Fig.8.The activation energy Ea, calculated from the slope of plot was found to be KJ/mol for adsorption of dyes on CDB. The negative values of Ea indicate the exothermic nature of the adsorption process. Table 2 indicates that the probability of dyes to stick on surface of CDB is very high $S^* << 1$ these values of Ea,S* confirm that, the adsorption process is physisorption.

Table 2. Thermodynamic parameters, activation energy and sticking probability of dyes in aqueous solution by CDB at 30°C

dyes	Fast Green FCF	Carmine	Rhodamine 6G	Safranine O
$-\Delta H$(kJ.mol-**1**)	6.701	9.190	8.061	2.297
ΔG(kJ.mol-**1**)	5.815	1.229	1.506	5.436
ΔS(J.mol-**1**)	41.30	34.38	31.57	25.52
-Ea(KJ.mol-1)	47.67	44.22	62.04	54.56
S*	0.58	0.70	0.51	0.60

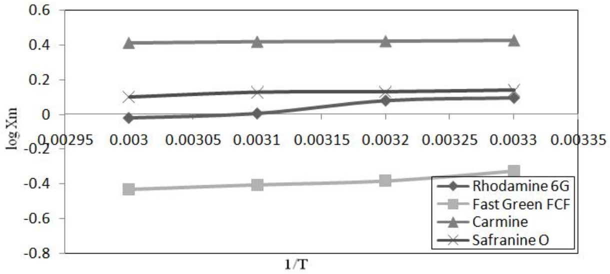

Fig.7. Relation between log Xm, 1/T for adsorption of dyes by CDB in 30°C

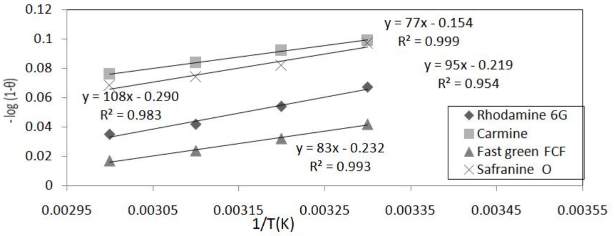

Fig.8. Relation between temperature and log(1-θ) for dyes sorption on CDB

Effect of initial dye concentration on adsorption of dyes

For performed this experimental, 0.2g portion of each dired adsorbent was treated with 20ml of dyes solution with concentration of (2-20mg/l) at PH 7 for 0.5h accompanied by mild shaking at room temperature. For.9 showed the effect of initial dye concentration (Co) (2-20mg/l) on the adsorption capacity by CDB. It was seen that the dyes adsorption efficiency increase with increase in its concentration and tends to attain saturation at higher concentration the percentage of dye removal decrease, the actual amount of dyes adsorbed per unit mass of CDB increased.

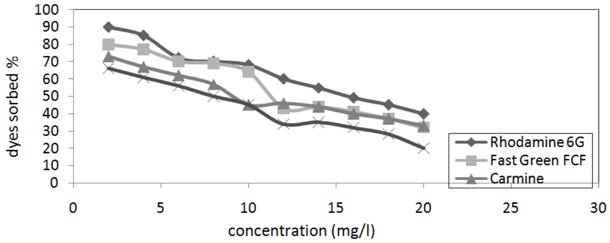

Fig.9. Influence of dyes concentration on CDB at 30°C

ADSORPTION KINETICS

In this study, the kinetic data of the CDB were analyzed using:

Pseudo-First –order-kinetic model

The Lagergren pseudo –first-order was defined as follows [26]

$$log(qe-qt) = log\ qe - \frac{K1t}{2.303}$$

where qe and qt are the amount of dyes adsorbed per unit weight of the adsorbent (mg/g) at equilibrium time and time t, respectively,K1 is the rate constant for the pseudo-first -order kinetics the values of log(qe-qt) were linearly correlated with t ,the plot of log(qe-qt) vs. t should give a linear relationship from which the values of K1 were determined from the slope of the plot Table 3. It many cases, the pseudo–first-order equation of Lagergen dosen't fit well with the whole range of contact time and is generally applicable over the initial stage of the adsorption process Fig.10. The calculated qe values aren't close to the experimental qe values which indicate that the adsorption of dyes onto CDB isn't a first order reaction.

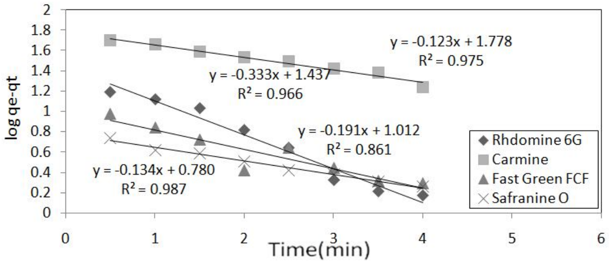

Fig.10. plot of the pseudo simple first order adsorption of dyes on CDB at 30ºC

Pseudo –second-order model

The pseudo –second –order model is represented by the following differential equation[27]

$$\frac{t}{qt} = \frac{1}{K2qe} + \frac{1}{qe}t$$

The slope and intercept of plot $\frac{t}{qt}$ vs. t were used to calculated the pseudo –second –order rate constant K_2 Fig.11.

Table 3. The values of K_1,K_2, the correlation coefficients and q_e at initial concentration 30°C

Co mg/l	Pseudo 1st order			Pseudo 2nd order		
	qe mgg^{-1}	K_1 min^{-1}	R^2	qe mgg^{-1}	K_2 gmg^{-1} min^{-1}	R^2
Fast Green FCF	0.988	5.235	0.861	6.024	3.921	0.975
Carmine	0.562	8.130	0.975	2.433	0.647	0.969
Rhodamine 6G	0.695	3.003	0.966	3.076	0.724	0.987
Safranine O	1.282	7.462	0.987	17.543	0.680	0.971

The values of equilibrium rate constant K_2 are present in Table 3. The correlation coefficients all examined data were found very high and calculated qe are almost near experimental qe values .This shows that the model can be applied for the entire adsorption process and confirms that the adsorption of dyes on CDB follows the pseudo –second –order kinetics model.

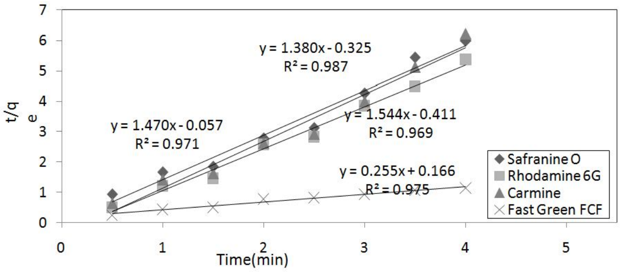

Fig.11. plot of pseudo second order adsorption of dyes on CDB at 30°C

Adsorption isotherms

Adsorption isotherms are important for the description of how adsorbates will interact with an adsorbent and are critical in optimizing the use of adsorbent[28]. Thus, the correlation of equilibrium data using either a theoretical or empirical equation is essential for adsorption data interpretation and prediction .Several mathematical models can be used to describe experimental data of adsorption isotherms Fig.12. Five famous isotherms equation, the Langmuir, Freundlich, Harkins-Jura, Tempkin and Dubinin-Radushkevich isotherms are applied in this study.

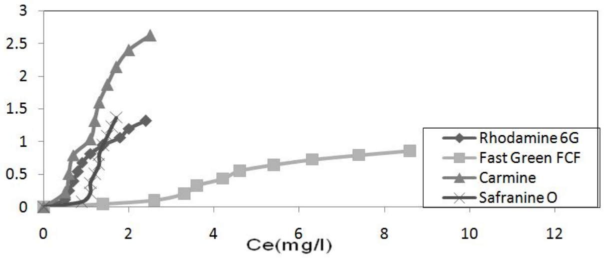

Fig.12 Adsorption isotherms of dyes solution on CDB at 30°C

Table 4. The Langmuir, Freundlich, Temkin, D-R and Harkins -jura constants for the adsorption of dyes solutions by CDB at 30ºC

Langmuir			Freundlich		Temkin		D-R		Harkins-jura	
dyes	K_L Lmg^{-1}	$-q_m$ mgg^{-1}	K_f mg$^{1-(1-n)}$ g^{-1}L$^{1/n}$	n	Ln kt Jmol^{-1}	Bt Lmg^{-1}	lnQm mol^{2}J^{-2}	k mgg^{-1}	B_2	A
Fast Green FCF	0.038	0.379	0.026	0.540	1.379	2.451	4.250	18.41	0.716	1.412
Carmine	0.658	2.754	0.993	0.760	3.553	7.278	2.597	9.216	1.276	0.533
Rhodamine 6G	0.386	1.855	0.542	0.753	1.992	4.713	2.933	4.999	1.122	0.447
Safranine O	0.065	0.106	0.185	0.227	2.061	4.090	2.265	3.649	1.054	0.365

The Langmuir isotherm is based on the assumption that the adsorption process takes place at specific homogenous sites within the adsorbent surface and that once a dye molecule occupies a site, no further adsorption can take place at that site, which concluded that the adsorption process is monolayer in nature. The Langmuir equation which is valid for monolayer adsorption onto a completely homogenous surface with a finite number of identical sites negligible interaction between adsorbed molecules, is represented in the linear form as follows[29]

$$\frac{Ce}{qe} = \left(\frac{1}{Klqm}\right) + \frac{1}{qm}Ce$$

Where Kl is the Langmuir adsorption constant (L/mg) and qm is the theoretical maximum adsorption capacity (mg/g). Fig.13 shows the Langmuir $\frac{Ce}{qe}$ vs. Ce plots for adsorption of dyes at 30°C. The value of Qm and Kl constants and the correlation coefficients for Langmuir isotherm are presented in Table 4.

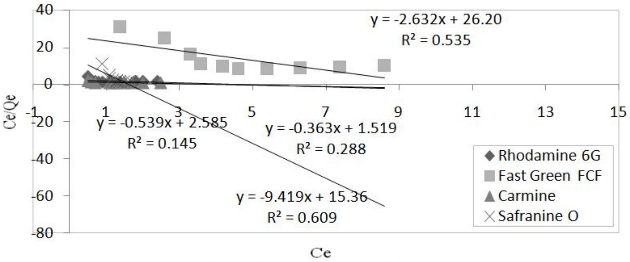

Fig.13:Langmuir linear for adsorption of dyes on CDB at 30°C

The Freundlich isotherm is derived by assuming a heterogeneous surface with a nonuniform distribution of adsorption heat over the surface was presented in the linear form as follows[30]

$$\log qe = \log Kf + \frac{1}{n}\log Ce$$

Where Kf(L/mg) and n is isotherm constants indicate the capacity and intensity of the adsorption, respectively. The $\frac{1}{n}$ factor also indicate heterogeneous factor Fig.14 shows the Freundlich log qe vs. log Ce plots for adsorption dyes at 30°C Table 4 shows the Freundlich adsorption isotherm constant and its respectively correlation coefficients. Heat of adsorption and the adsorbent–adsorbate interaction on adsorption isotherms were studied by tempkin and its equation is given as [31]

$$qe = Bt\ln Kt + Bt\ln Ce$$

Where Bt=RT/bT, T is the absolute temperature in k, Kt the equilibrium binding constant (L/mg) and Bt is related to the heat of adsorption. Fig.15 shows the tempkin qe vs. ln Ce plots for adsorption of dyes at 30°C. The constants obtained for Tempkin isotherm are shown in Table 4.

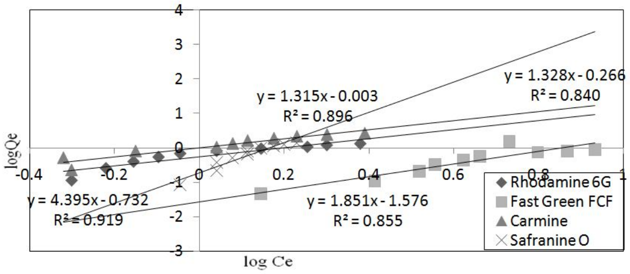

Fig.14. Freundlich linear for adsorption of dys on CDB in 30°C

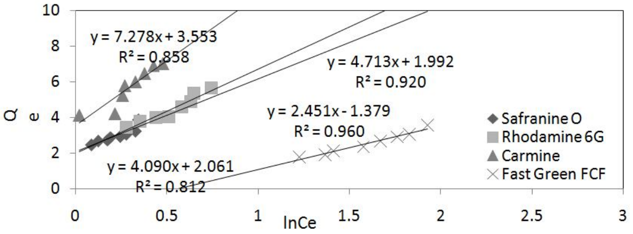

Fig.15. TemPkin linear for adsorption of dyes on CDB at 30°C

The linear form of Dubinin-Radushkevich isotherm equation can be expressed as[32]

$$\ln qe = lnQm - K \in^2$$

Where Qm is the theoretical monolayer saturation capacity (mg/g), K is the Dubinin-Radushkevich model constant ($mol^2.Kj^{-2}$), Σ is the Polanyi potential and is equal to:

$$\in = RT\ ln\left(1 + \frac{1}{Ce}\right)$$

The plot of ln qe vs. $\in^2$ at different temperature for dyes is present in Fig16.The constant obtained for D-R isotherm are shown in Table 4.

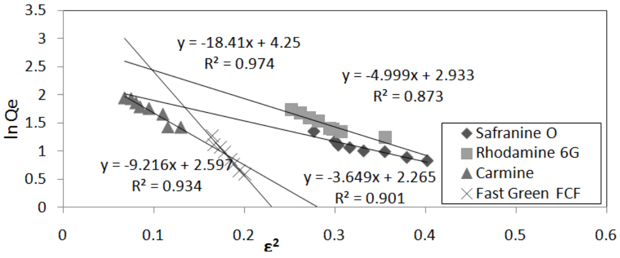

Fig.16. Dubinin-Radushavich linear for adsorption of dyes on CDB at 30°C

The Harkins –Jura adsorption can be expressed [33]

$$\frac{1}{qe^2} = \frac{B_2}{A} - \frac{1}{A}\log Ce$$

Where B_2 and A are the isotherm constant. The Harkins –Jura adsorption accounts to multilayer adsorption and can be explained with the existence of a heterogeneous pore distribution $\frac{1}{qe^2}$ was plotted vs. log Ce Fig.17. Isotherm constants and correlation coefficients are summarized in Table 4.

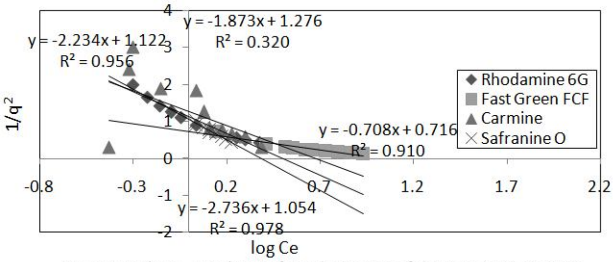

Fig.17: Harkins -Jura linear for adsorption of dyes on CDB at 30°C

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