

## KINETIC MODELING AND ISOTHERM STUDIES ON A BATCH REMOVAL OF ACID RED 114 BY AN ACTIVATED PLANT BIOMASS

N. RAJAMOHAN<sup>1,\*</sup>, M. RAJASIMMAN<sup>2</sup>, R. RAJESHKANNAN<sup>2</sup>,  
B. SIVAPRAKASH<sup>2</sup>

<sup>1</sup>Department of Chemical Engineering, Sohar University, Sohar, Oman

<sup>2</sup>Department of Chemical Engineering, Annamalai University, Annamalainagar, India

\*Corresponding Author: natrajmohan@gmail.com

### Abstract

In this paper, the dye Acid Red 114 (AR 114) was removed from aqueous solutions using Acid-Activated *Eichhornia Crassipes* (AAEC) under batch conditions. The optimum conditions for AR 114 removal were found to be pH 1.5, adsorbent dosage = 1.25 g/L of solution and equilibrium time = 3 h. The equilibrium data were evaluated for compliance with Langmuir, Freundlich and Temkin isotherms and Langmuir isotherm was found to fit well. The maximum sorption capacity was estimated as 112.34 mg/g of adsorbent. Also, adsorption kinetics of the dye was studied and the rates of sorption were found to follow pseudo-second order kinetics with good correlation ( $R^2 \geq 0.997$ ). The kinetic study at different temperatures revealed that the sorption was an endothermic process. The activation energy of the sorption process was estimated as 9.722 kJ/mol.

Keywords: *Eichhornia Crassipes*, Dye, Sorption, Isotherms.

### 1. Introduction

The increasing demand for efficient and low-cost treatment methods for removal has given rise to the identification of low cost alternative adsorbents. An alternative technology which is gaining tremendous importance for the removal of dyes from its aqueous solutions is biosorption. This process involves the utilization of the living materials to adsorb the dyes from waste waters either by pure physico-chemical pathways or metabolic mediation [1].

Textile industries are characterized by high consumption of water and use a wide variety of dyes which contains carcinogenic and mutagenic chemicals like benzidine, metals, etc. [2]. Industrial effluents containing dyes pose a serious threat

**Nomenclatures**

$A$	Activation energy, kJ/mol
$B$	Heat of adsorption
$C_e$	Concentration of the dye at equilibrium
$C_o$	Initial concentration of the dye
$E_a$	Arrhenius factor, g/mg.min
$I$	Constant that is related to boundary layer thickness, mg/g
$K_f$	Freundlich constant, mg/g
$K_L$	Langmuir constant, L/mg
$K_T$	Equilibrium binding constant
$k$	Rate constant
$k_t$	Intra particle diffusion rate constant, mg/(g min <sup>1/2</sup> )
$k_2$	Equilibrium rate constant for pseudo-second order adsorption, g/mg.min
$M$	Mass of the adsorbent, g
$q_e$	Amount of dye adsorbed on the adsorbent, mg/g
$q_{max}$	Maximum adsorption capacity, mg/g
$q_t$	Amount of dye adsorbed at time, $t$ , mg/g
$q_1$	Maximum adsorption capacity, mg/g
$R$	Gas constant
$T$	Solution temperature, K
$V$	Volume of the solution, L
$1/n$	Adsorption intensity

to the environment because of its high toxicity and possible accumulation. Moreover, these synthetic dyes have complex structure and aromatic rings which make them more difficult to be degraded. Various conventional physico-chemical methods available for dye removal are coagulation, oxidation, ozonation, adsorption, biological and electro chemical processes [3-8]. Because of the presence of large organic content and complex molecular structure, most of the conventional methods are inefficient in removing the dyes. Also, these processes are expensive, require large land area and cannot be effective for dye waste water treatment [9]. Even though activated carbon is an effective sorbent; it suffers from limitations like high operating costs, high price and difficulty with regeneration. These disadvantages resulted for the search for a cheaper adsorbent.

A number of researchers have demonstrated that some low-cost adsorbents rice husk [10], mango seed kernel powder [11], bagasse pith [12], modified tea waste [13], regenerated clay [14] and other agricultural biomass [15] for removal of dyes. However, the adsorption capacities of the above adsorbents are not very high; In order to improve the efficiency of the adsorption processes, it is necessary to develop cheap and easily available adsorbents with high adsorption capacities. Eichornia Crassipes (commonly called water hyacinth) is a wild fern belonging to the Family Pontederiaceae. It is a submerged aquatic plant which invades the waterways abundantly. It grows very rapidly and is considered as an unwanted plant since it blocks navigation and interferes with irrigation and power generation [1]. Also, this plant forms mats which prevent the penetration of sunlight and reduce the aeration of water resulting in the decrease of dissolved oxygen. Mechanical collection and dumping, used as a method for disposal of this

aquatic plant, is not economical on large scale. In this study, Acid Activated Eichornia Crassipes (AAEC) was utilized to remove the anionic dye (Acid red 114) from its aqueous solution.

The objective of this study was to study the effect of operating parameters like initial dye concentration, sorbent dosage, contact time and temperature on the sorption efficiency. In addition, kinetic studies were conducted to determine the contact time required to reach equilibrium. The equilibrium studies were also performed.

## 2. Experimental and Methods

### 2.1. Biosorbent preparation

The *Eichornia Crassipes* (water hyacinth) roots used in this study were obtained from a pond near by Department of Chemical engineering, Annamalai University, Annamalainagar, Tamilnadu, India. The collected biomaterial was extensively washed with distilled water to remove soil and dust and sliced into small pieces. The sliced material was dried by exposure to the sunlight for 3 days and subsequently at 60°C for 48 h in a hot air convection oven. The dried material was milled into a powder using blender and sieved to obtain constant size particles (between 250 and 400 µm).

### 2.2. Activation of the sorbent

The sieved powder of the raw biosorbent (10 g) was soaked in 100 mL of 4N HCl in a mechanical shaker at 200 rpm for 24 h. The samples were filtered and rinsed with distilled water, until the pH was neutral. The treated material was dried in sunlight and sealed in plastic bags and stored in desiccators for further use.

### 2.3. Preparation of aqueous dye solutions

The dye, Acid red 114 ( $C_{37}H_{28}N_4O_{10}S_3Na_2$ ), was purchased from Sigma-Aldrich (India) and used without any purification. All other reagents were of analytical reagent grade and were obtained from Merck chemicals (India). A calculated amount of the dye was dissolved separately in 1 L of deionized water to prepare stock solutions (1000 mg/L), which were kept in dark coloured glass bottles. The physical and chemical characteristics of the dye were presented in Table 1.

**Table 1. Physico-Chemical Characteristics of the Dye.**

Generic Name	C.I. Acid Red 114
Abbreviation	AR 114
Commercial name	Acid Red 114
Dye type	Anionic
Chromophore	Diazo
Chemical formula	$C_{37}H_{28}N_4O_{10}S_3Na_2$
Molar mass(g/mol)	830
$\lambda$ max(nm)	524
Color	Dark red
Colour Index number	23,635
Physical state	Dry powder

## 2.4. Kinetic parameters

The kinetic studies were conducted to estimate the contact time required for the attainment of equilibrium between the dissolved and solid bound adsorbate. For the first set of experiments, different sorbent dosages (such as 0.25, 0.5, 0.75, 1.0, 1.25, 1.5, 1.75 and 2.0 g/L) was mixed thoroughly with 100mL of 100 mg/L dye solution and suspensions were placed in a mechanical shaker operated at a constant speed of 200 rpm and room temperature (30°C). The samples were collected at regular space interval and centrifuged at 8000 rpm for 5 min. The colour reduction profiles were obtained using the absorbance measurements. The adsorbent and adsorbate Samples were then analyzed for reduction in absorbance using UV/V spectrophotometer (Shimadzu-UV-2101, Japan). Similar experiments were carried out by agitating with 100mL of dye solutions whose concentrations were 75, 150 and 200 mg/L at the optimum pH with 1.25 g/L of AAEC in a mechanical shaker operated at room temperature. In order to identify the adsorption mechanism, the rate constants for chemisorption and intraparticle diffusion for AR 114 were determined using the models for a pseudo-second-order [16] and intraparticle diffusion [17].

The pseudo-second-order kinetic model is expressed as

$$\frac{t}{q_t} = \frac{1}{k_2 q_1^2} + \frac{1}{q_1} t \quad (1)$$

where  $q_1$  (mg/g) is the maximum adsorption capacity and  $q_t$  (mg/g) is the amount of dye adsorbed at time,  $t$ , and  $k_2$  (g/mg.min) is the equilibrium rate constant for pseudo-second order adsorption.

The intraparticle diffusion kinetic model is expressed as

$$q_t = k_t t^{1/2} + I \quad (2)$$

where  $q_1$  (mg/g) is the maximum adsorption capacity and  $q_t$  (mg/g) is the amount of dye adsorbed at time,  $t$ , and  $k_t$  (mg/(g min<sup>1/2</sup>)) is the intraparticle diffusion rate constant and  $I$  (mg/g) is a constant that is related to boundary layer thickness.

## 2.5. Equilibrium studies

The effect of initial pH of solution on the amount of dye adsorbed onto AAEC was studied in the pH range from 1.5 to 10.5 by the addition of 0.1N HCl or 0.1 N NaOH. In this study, 50 mL of 100 mg/L dye solution was agitated with 0.1g of AAEC at room temperature (30°C) and at a constant speed of 200 rpm for 10 h.

Langmuir, Freundlich and Temkin models, Eqs. (3), (4) and (5) respectively were employed to fit the experimental data.

$$\frac{c_e}{q_e} = \frac{c_e}{q_{max}} + \frac{1}{K_L q_{max}} \quad (3)$$

$$\ln q_e = \ln K_f + \frac{1}{n} \ln c_e \quad (4)$$

$$q_e = B \ln K_T + B \ln c_e \quad (5)$$

where  $q_e$  (mg/g) and  $C_e$  (mg/L) are the amount of AR 114 adsorbed on the adsorbent and concentration of the dye at equilibrium, respectively;  $q_{max}$  (mg/g) is the maximum adsorption capacity and  $K_L$  (L/mg) is the Langmuir constant related to the free energy of adsorption,  $K_f$  (mg/g) is the Freundlich constant which is an estimate of relative adsorption capacity of the sorbent and  $1/n$  is the

adsorption intensity.  $B (=RT/b)$  is the heat of adsorption and  $K_T$  (L/mg) is the equilibrium binding constant. The amount of dye adsorbed during the contact time with AAEC was estimated using the following equation

$$q_e = \frac{V(C_0 - C_e)}{M} \quad (6)$$

where  $C_0$  is the initial concentration of the dye,  $V$  is the volume of the sample solution and  $M$  is the mass of the adsorbent.

## 2.6. Activation energy determination

The feasibility and spontaneous nature of the adsorption process is reflected by the thermodynamic parameters. Arrhenius equation, Eq. 7, represents the relationship between rate constant and temperature and is given as:

$$\ln k = \ln A - (E_a/RT) \quad (7)$$

where  $k$  is the rate constant obtained at different temperatures with the best fit kinetic model,  $E_a$  (kJ/mol) is the activation energy,  $A$  (g/mg.min), is the Arrhenius factor,  $R$  is the gas constant (8.314 J/mol K) and  $T$ , (K) is the solution temperature.

## 3. Results and Discussion

### 3.1. Effect of pH of the solution

The adsorption capacity and percentage dye removal decreased with the increase in initial solution of the pH as shown in Fig. 1. The maximum dye uptake was observed to be 76 mg/g at pH 1.5. The adsorbent used, *Eichhornia Crassipes*, is composed of alcoholic hydroxyl groups [18], which could be affected by the pH of the solution. In this regard, the possible reasons identified for this effect of pH are organic properties of the sorbent, dye molecule structure and electrostatic attraction. Better removal at low pH values are attributed to the presence of higher electrostatic attractions between anionic dye molecules and the positively charged surfaces of the adsorbent. All acid dyes release coloured dye anions into the solution. The surface charges of the sorbent, which is influenced by the hydrogen ions present, were found to be responsible for the sorption of the anionic sorbate molecules. Identical observations were reported for the sorption of acid red 114 using activated carbon produced from seed shells [9] and direct dyes by soy meal hull [19]. Requirement of highly acidic environment could be a limitation with this adsorbent.

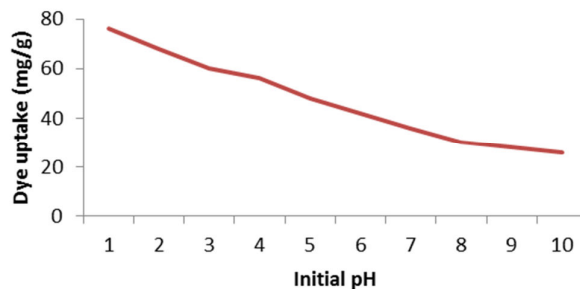


Fig. 1. Effect of Initial pH of the Solution for the Sorption of AR 114 ( $t=3$  h;  $C_0=100$  mg/L,  $M=1.25$  g/L).

### 3.2. Effect of initial concentration

The effect of initial concentration AR 114 concentration on the kinetics of dye sorption was studied at optimal pH (1.5), AAEC-dosage (1.25 g/L) and 30°C in the dye concentration range of 50-200 mg/L. An increase in the initial concentration of dye resulted in increase in removal percentages and dye uptake and is represented in Fig. 2. As the initial concentration changed from 50 mg/L to 200 mg/L the equilibrium dye uptake increased from 62 mg/g to 86 mg/g. Similar results are reported for the study on sorption of the dye, Disperse Orange 25, onto activated carbon from a plant biomass [2]. During the experimental studies, it was observed that majority of the dye was removed in the initial phase where the dye uptake is rapid. The percentage removal during the first 45 min was observed to be 58%, 74%, 79% and 84% respectively for initial dye concentrations of 50, 100, 150 and 200 mg/L. The reason attributed to this effect is the presence of higher concentration gradient in the initial phase of the sorption process [1].

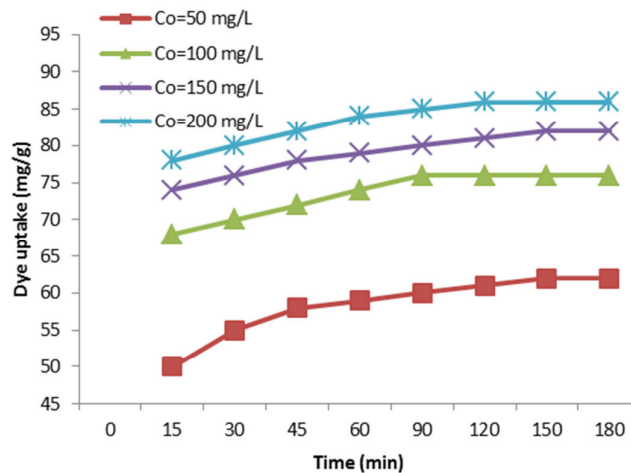


Fig. 2. Effect of Initial Concentration of the Dye for the Sorption of AR 114 ( $t=3$  h,  $pH=1.5$ ,  $M=1.25$  g/L).

### 3.3. Effect of sorbent dosage

Adsorbent dosage is identified as a vital parameter influencing the uptake of dye. Specific surface area which depends on the sorbent dosage is an important factor that affects the sorptive capacity [20]. The change in the percentage dye removal and uptake at various sorbent dosages (0-2 g/L) was studied by keeping the other process variables constant. An increase in the percentage dye removal was observed with increase in sorbent dosages, as shown in Fig. 3. This was because of the availability of more surface area for sorption through large availability of functional groups [9]. But, the adsorbent dosage was found to have a negative effect on dye uptake, which decreases with increase in sorbent dosage. Aggregation effect of sorbent particles was found to occur at higher dosages. This in turn reduced the total surface area and increased the length of the diffusion path, which led to a decrease

in dye uptake [21]. The relationship between the amount of dye adsorbed at equilibrium,  $q_e$ , and the adsorbent dosage,  $M$ , is given by

$$q_e = 29.863 \ln(M) + 37.54 \quad (8)$$

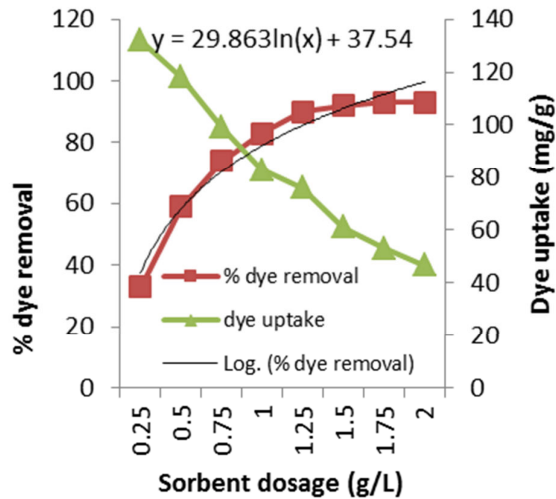


Fig. 3. Effect of Sorbent Dosage on Percentage Dye Removal and Dye Uptake for the Sorption of AR 114 ( $t = 3$  h,  $\text{pH} = 1.5$ ,  $C_0 = 100$  mg/L).

### 3.4. Effect of operating temperature

The equilibrium sorption capacity of AR 114 increased with increase in temperature and is shown in Fig. 4. Thus, the sorption process is identified as an endothermic process which always favour higher temperatures.

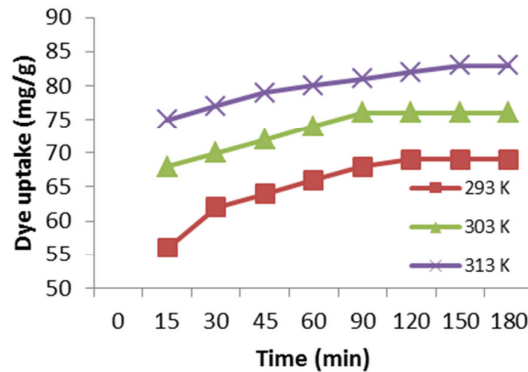


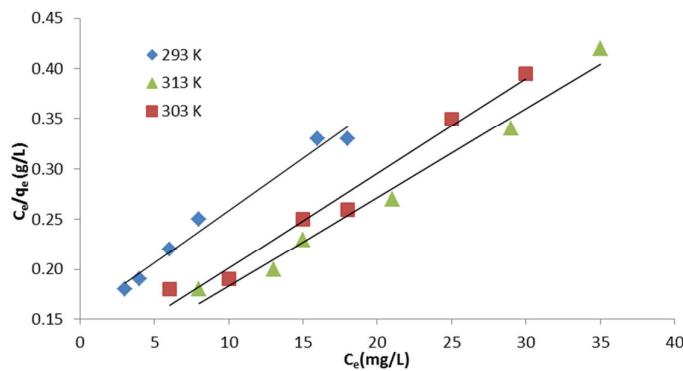
Fig. 4. Effect of Temperature on Dye Uptake for the Sorption of AR 114 ( $t = 3$  h,  $\text{pH} = 1.5$ ,  $C_0 = 100$  mg/L).

### 3.5. Biosorption equilibrium

The Langmuir, Freundlich isotherms are the most frequently used two parameter models in the literature describing the non-linear equilibrium between amount of dye adsorbed on the acid treated water hyacinth roots ( $q_e$ ) and equilibrium concentration of solution ( $C_e$ ) at a constant temperature (30°C). The linearized forms of the models, Eqs. (3), (4) and (5), are used for the determination of isotherm constants. Table 2 shows the values of the isotherm constants along with the respective correlation coefficients. From the experimental results, it was observed that Langmuir model fits the data very well. The Langmuir constants;  $q_{max}$  and  $K_L$ , are calculated from the plot of  $C_e$  versus  $C_e/q_e$ . Figure 5 shows the Langmuir adsorption isotherms of the AR 114 onto AAEC. The maximum adsorption capacity was estimated as 112.34 mg/g at 313 K. All the isotherm model parameters were tabulated in Table 2.

**Table 2. Isotherm Constants.**

Temp. (K)	Langmuir			Freundlich			Temkin		
	$q_{max}$ (mg/g)	$K_L$ (L/mg)	$R^2$	$n$	$K_f$ (L/g)	$R^2$	$K_T$ (L/mg)	$B$	$R^2$
293	92.6	0.07	0.98	2.63	12.12	0.973	1.329	21.116	0.969
303	106.4	0.087	0.98	3.195	18.8	0.914	2.772	11.722	0.8731
313	112.34	0.094	0.988	4.005	29.6	0.97	3.857	9.54	0.961



**Fig. 5. Langmuir Plot for the Sorption of AR 114 on AAEC.**

The essential characteristics of Langmuir isotherm can be expressed in terms of  $R_L$ , a dimensionless separation factor of equilibrium parameter, which is defined as [2]

$$R_L = \frac{1}{1 + K_L C_0} \quad (9)$$

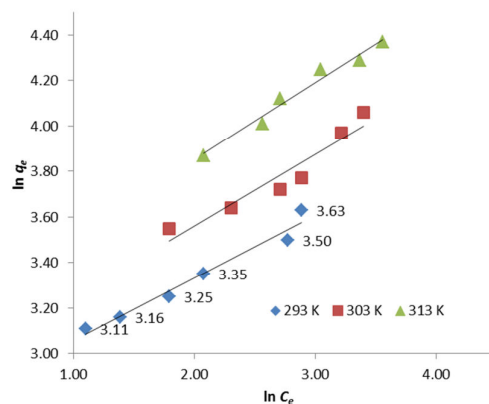
The  $R_L$  values calculated were found to be between 0.096 and 0.125 at different concentrations indicating favourable adsorption of dye on AAEC [9, 22]. The sorption capacity observed in this study is comparable with the capacities reported on various literatures on sorption of AR-114 and a comparison was presented in Table 3.



**Table 3. Comparison with the Sorption Capacities of AR 114 from the Literature by Various Adsorbents.**

Adsorbent	Adsorption capacity, (mg/g)	Source
Acid activated <i>Eichornia Crassipes</i>	112.34	This study
Filtrisorb F 400	103.5	Choy et al., 2000
Activated carbon-charcoal	101	Choy et al., 1999
Bagasse pith(raw)	20	Chen et al., 2001
Activated carbon from cotton seed shell	153.85	Thinakaran et al., 2008 [9]
Activated carbon from gingelly seed shell	102.04	Thinakaran et al., 2008 [9]

The Freundlich isotherm equation was applied to the equilibrium data and the Freundlich constants,  $K_f$  and  $1/n$  are calculated from the plot between  $\ln C_e$  versus  $\ln q_e$  and is shown in Fig. 6. The values of the Freundlich constants are strongly affected by change in temperature and a direct proportionality was found to exist between the constants and temperature. The values of  $K_f$  was observed to increase from 12.2 to 29.6 L/g and  $n$  increased from 2.63 to 4.0 (when the temperature increased from 293 to 313 K). The sorption capacity of AAEC used in this study was found to be many times higher than that of raw bagasse pith. Acid activation could be the reason for the better performance of the selected adsorbent. In addition, the carbon content of bagasse pith could be comparatively less.

**Fig. 6. Freundlich Plot for the Sorption of AR 114 on AAEC.**

This fact indicates the increasing intensity of adsorption with increase in temperature. Similar observations were reported on the removal of dye Disperse Orange 25 using activated carbon from *E.rigida* [2]. Temkin isotherm constants were evaluated from the plot of  $\ln C_e$  versus  $q_e$ , Fig. 7, and the value of  $K_T$ , the equilibrium constant, was found to increase from 1.329 to 3.857 when the temperature increased from 293 K to 313 K. Thinakaran et al. have reported a similar trend in their studies on removal of dye AR 114 by activated carbons from seed shells.

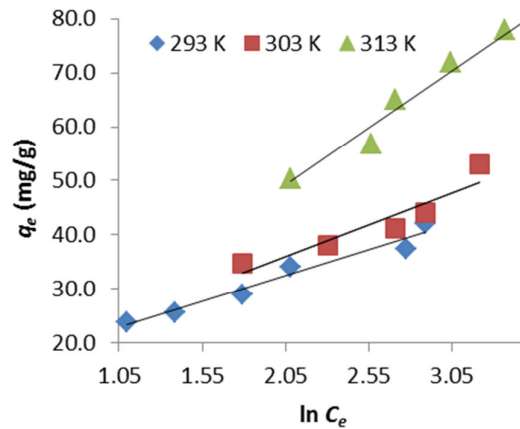


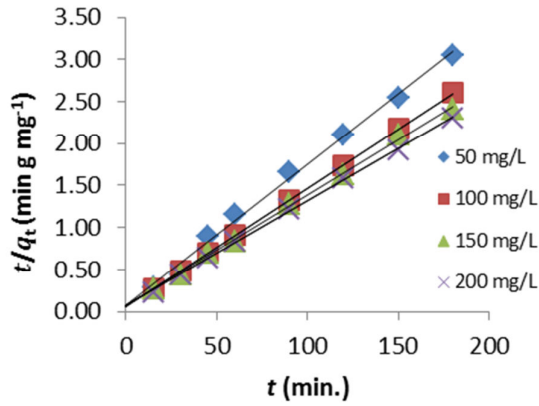
Fig. 7. Temkin Plot for the Sorption of AR 114 on AAEC.

### 3.6. Kinetic studies

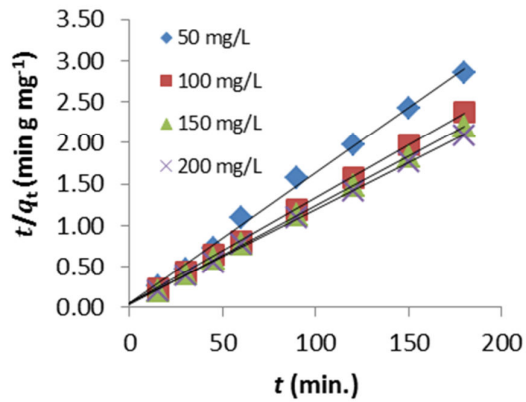
The kinetic models, namely pseudo-second-order and intra-particle diffusion models were used to analyse the transient behavior of the dye sorption process. The equilibrium uptakes were predicted by the rate constants and the corresponding correlation coefficients were summarized in Table 4. Values of  $k_2$  and  $q_1$  were found from the plot of  $t/q_t$  versus  $t$ , Fig. 8. An increase in the temperature led to an increase in  $q_1$  values. However, the pseudo-second-order model cannot identify the diffusion mechanism. Intra-particle diffusion model was employed to identify the mechanism by plotting the uptake,  $q_t$ , versus the square root of time,  $t^{1/2}$ , Fig. 9. If the plot is linear with the line passing through the origin, then intra particle diffusion is the rate controlling step [23]. The constants in the intra particle diffusion model,  $K_s$ , and  $I$  were evaluated from Fig. 9. The correlation coefficients for the pseudo-second-order kinetic model were found to be higher than that of the intra particle diffusion model. These results confirmed that the actual adsorption contains both surface adsorption and intra particle diffusion [2].

Table 4. Pseudo-second-order and Intra-particle Diffusion Model Constants.

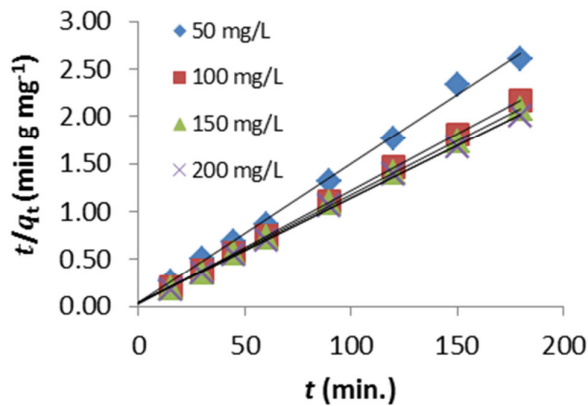
	Initial Dye Concentration (mg/L)				Initial Dye Concentration (mg/L)				Initial Dye Concentration (mg/L)			
	50	100	150	200	50	100	150	200	50	100	150	200
Pseudo second order												
$k_2 \times 10^3$ (g/mg min)	4.19	3.36	2.64	2.38	4.7	3.48	3.12	2.6	5.6	4.33	3.42	3.1
$q_1$ (mg/g)	59.52	70.92	75.76	80	62.9	78.12	84.03	87.72	68.5	84.03	89.1	91
$R^2$	0.998	0.999	0.997	0.999	0.996	0.999	0.999	0.999	0.999	0.999	0.999	0.998
Intraparticle diffusion												
$k_t$ (mg/g min <sup>1/2</sup> )	1.57	1.923	1.526	1.270	1.74	1.587	1.139	1.095	1.65	1.112	0.897	0.7536
$I$	39.14	46.32	56.41	61.9	40.89	57.25	67.67	71.80	48.46	69.53	75.16	79.79
$R^2$	0.982	0.873	0.943	0.982	0.958	0.945	0.969	0.931	0.924	0.927	0.838	0.866



(a) 293 K

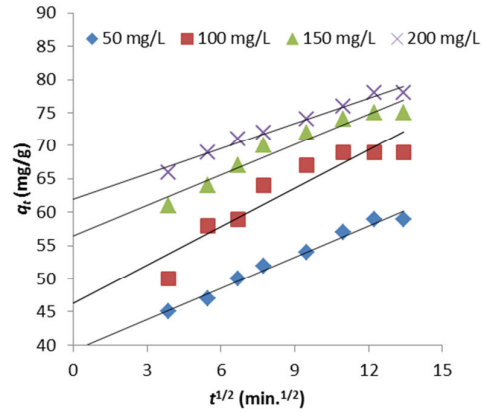


(b) 303 K

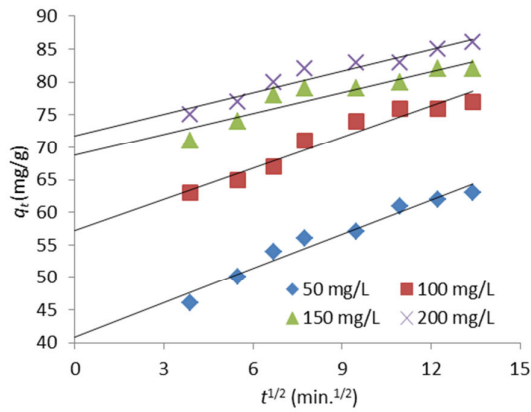


(c) 313 K

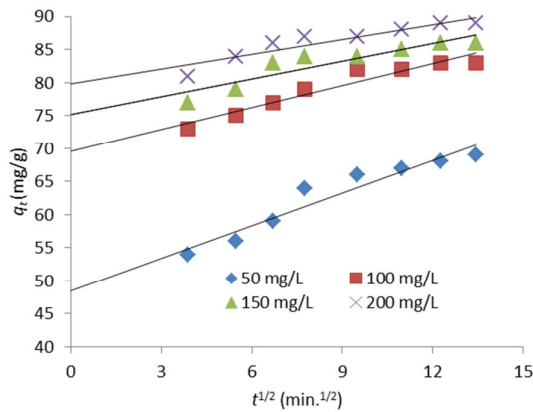
Fig. 8. Pseudo-second-order Kinetic Plots for the Sorption of AR 114 on AAEC at (a) 293 K, (b) 303 K and (c) 313 K.



(a) 293 K



(b) 303 K

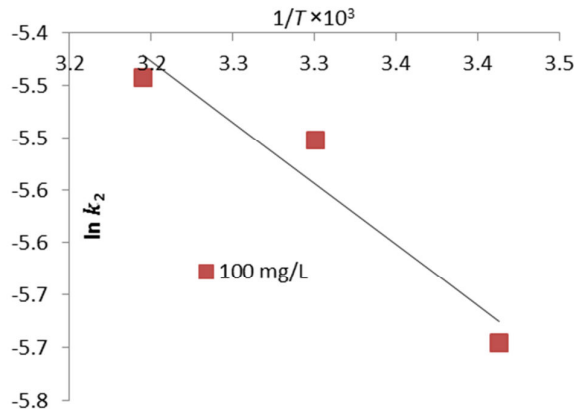


(c) 313 K

Fig. 9. Pseudo-second-order Kinetic Plots for the Sorption of AR 114 on AAEC at (a) 293 K, (b) 303 K and (c) 313 K.

### 3.7. Activation energy of adsorption

Arrhenius equation, Eq. 7, is used for the determination of activation energy for the adsorption of AR 114 on AAEC. The rate constant from the best fit model, pseudo-second-order kinetic model,  $k_2$ , was used to make a plot of  $\ln k_2$  versus  $1/T$ , Fig. 10. The slope of the best fit line revealed the Activation energy ( $E_a$ ) of the biosorption process to be 9.722 kJ/mol. The value of  $E_a$  indicates the energy barrier during adsorption [24]. The value of  $E_a$  calculated in this study is comparable with the values reported in literature [25]. This indicates that the biosorption of AR 114 by AAEC is chemisorption in nature. This fact is consistent with the properties of the adsorbent and Langmuir-type adsorption isotherm [18].



**Fig. 10. Determination of Activation Energy by Arrhenius Plot [ $C_0=100$  mg/L].**

### 4. Conclusions

The modified *Eichhornia Crassipes* proved to be an effective adsorbent for the removal of the selected dye, AR 114. The dye uptake was found to be strongly influenced by pH. Inverse proportionality is observed between the sorbent dosage and dye uptake. Langmuir isotherm represented the equilibrium sorption data very well. The pseudo-second-order kinetic model fitted well for the experimental data and revealed a rapid sorption rate. Endothermic nature of this sorption process is proved by the values of the rate constants at different operating temperatures. The activation energy for this sorption process was estimated as 9.722 kJ/mol.

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