



*Original Article*

## Bottom ash adsorption of basic dyes from their binary aqueous solutions

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### Abstract

The uses of low-cost adsorbent have been investigated as a replacement for the current expensive methods of removing dyes from aqueous solution. As such bottom ash is used as a low-cost adsorbent. The prepared adsorbent was characterized and used for the removal of three basic dyes namely Malachite green (MG), Methylene blue (MB), and Crystal violet (CV) in binary systems. Batch adsorption experiments were conducted to determine the effect of contact time and adsorbent dosage. The adsorption data is fitted with the linear forms of the Langmuir and Freundlich models. The results indicated that the Freundlich adsorption isotherm fitted the data better than the Langmuir adsorption isotherm for all systems. Pseudo-first-order and pseudo-second-order constant were determined. The results of the study demonstrated that bottom ash could be used as an effective low cost adsorbent for the removal of basic dyes from aqueous solution.

**Keywords:** bottom ash, adsorption, kinetics, isotherms, binary system

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### 1. Introduction

The huge growth in the textile industries has resulted in an immense increase in the complexity and volume of dye containing wastewater (Kadam *et al.*, 2013). Worldwide annual textile production is currently 30 million tons with expected growth of 3% per annum (Walker and Weatherley, 1997). Today there are more than 10,000 dye types available commercially (Nigam *et al.*, 2000). Many industries, such as textile, paper, printing, leather, food, cosmetics, etc., use dyes to color their products, which generate a considerable amount of colored wastewater (Monash and Pugazhenthii, 2009). This colored wastewater may mix with surface-groundwater systems and thus pollute the drinking water. Typical textile industry wastewater characteristics can be summarized by a COD range from 150 to 12000 mg/L, total suspended solids between 2900 and 3100 mg/L, total Kjeldahl nitrogen from 70 to 80 mg/L, and BOD range from 80 to 6000 mg/L leading to a BOD/COD ratio of around 0.25, showing that it

contains large amounts of non-biodegradable organic matter (Oller *et al.*, 2011). The consumption of this polluted water is a threat to human health due to either toxic or mutagenic and carcinogenic nature of dyes (Gong *et al.*, 2005). Dyes even in low concentration are visually detected and affect the aquatic life and food web (Muthuraman and Ibrahim, 2013). Water with high amount of color is not suitable for cloth washing, dyeing, paper industry, beverages production, dairies and other food products, textile industry, or plastic production (Malakootian and Fatehizadeh, 2010). Moreover, they are unusually resistant to degradation due to their complex structure and xenobiotic properties (Cristovao *et al.*, 2011; Gupta *et al.*, 2007). Hence, removal of dyes from such wastewaters is a major environmental problem and complete dye removal is necessary (Papić *et al.*, 2004).

Generally, the treatments of the dyestuff effluents can be classified into three major categories, namely, biological, chemical and physical treatment. Each of the above treatment methods have their own merits and demerits and are reported in many literatures (Crini, 2006; Garg *et al.*, 2004). Adsorption is one of the most effective physical processes for colour removal (Filipkowska *et al.*, 2002). Even though activated carbon showed advantages, the main drawback of activated

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carbon is the cost and difficulty in regeneration (Liu *et al.*, 2007). Therefore, in recent years, this has prompted a growing research interest in the production of activated carbons from renewable and cheaper precursors which are mainly industrial and agricultural by-products (Tan *et al.*, 2008). Low cost adsorbents such as coal fly ash (Hsu, 2008), bagasse fly ash (Rachakornkij *et al.*, 2004), coir pith (Ramesh *et al.*, 2011a,b) red mud (Gupta *et al.*, 2004), de-oiled soya (Mittal *et al.*, 2005, 2008, 2013), hen feathers (Mittal, 2006; Mittal *et al.*, 2012, 2013), eggshell waste (Darai *et al.*, 2013), cyperus rotundus (Ramesh *et al.*, 2013) etc. were used to remove several hazardous pollutants from aqueous solution.

In the present study, attempts have been made to utilize bottom ash, an industry waste, as a low-cost adsorbent for the removal of three basic dyes- Crystal violet (CV), Methylene blue (MB) and Malachite Green (MG) in binary systems from aqueous solution in batch mode. Bottom ash has been found as a good adsorbent for removing various pollutants such as crystal violet (Mittal *et al.*, 2010; Nidheesh *et al.*, 2011, 2012a, b; Gandhimathi *et al.*, 2012), fluoride (Ramesh *et al.*, 2012), malachite green (Gupta *et al.*, 2004; Gandhimathi *et al.*, 2012), methyl violet (Mittal *et al.*, 2008), rose bengal (Gupta *et al.*, 2012), methylene blue (Gandhimathi *et al.*, 2012) etc. The objectives of this paper are to report the effect of various process parameters on the extent of removal of dyes, to optimize the process parameters and to apply various adsorption isotherms and kinetic equations to the adsorption data.

## 2. Materials and Methods

### 2.1 Adsorbent

The thermal power plant waste material, 'bottom ash' was utilized as potential adsorbent in the study. The adsorbents were obtained from Neyveli Lignite Corporation Limited, Neyveli.

### 2.2 Characterization of adsorbent

The pH value of the adsorbents were determined by mixing 2 g of adsorbent with 100 mL of distilled water and recording pH at every 1 h interval for a period of 24 h. The loss on ignition (LOI) was determined by heating a pre-weighed dry sample (left at 105°C in an oven and then cooled in desiccators) to 600°C over a period of 1 h (Bayat, 2002). Fourier Transform Infra Red (FTIR) spectroscopic techniques were utilized to identify their functional groups of adsorbent. FTIR spectra of the samples were also recorded on PerkinElmer Model System 2000 using the KBr pellet method.

The Point of Zero Charge (PZC) was determined using the solid addition method (Oladoja and Aliu, 2009). 45 mL of 0.1M KNO<sub>3</sub> solution was transferred in series of 100 mL conical flasks. The pH values of the solution were roughly adjusted from 2 to 10 by adding either 0.1N HNO<sub>3</sub> or NaOH (pH<sub>0</sub>). The total volume of the solution in each flask was made

exactly to 50 mL by adding the KNO<sub>3</sub> solution. 1 g bottom ash was added to each flask and the suspension was manually agitated. The pH values of the supernatant liquid were noted (pH<sub>f</sub>). The difference between the initial and final pH values ( $\Delta\text{pH} = \text{pH}_0 - \text{pH}_f$ ) was plotted against the pH<sub>0</sub>. The point of intersection of the resulting curve at  $\Delta\text{pH}$  becomes zero will outcomes the value of PZC.

In order to provide a better understanding of the environmental impacts of adsorbent utilization, the solubility characteristics of various chemical species associated with bottom ash were examined. One gram sample of adsorbent was equilibrated with 200 mL of distilled water for 2 h (Bayat, 2002). After filtration, the major chemical constituents in the filtrate were measured. The measurement of heavy metal concentration was done using an S Series Atomic Absorption Spectrophotometer (AAS).

### 2.3 Adsorbate

The dyestuffs were used as the commercial salts. 1. Methylene Blue (C.I. 52015,  $\lambda_{\text{max}} = 663 \text{ nm}$ , M.W = 373.9 g/mol, M.F = C<sub>16</sub>H<sub>18</sub>ClN<sub>3</sub>S 3H<sub>2</sub>O), 2. Malachite Green (C.I. 42000,  $\lambda_{\text{max}} = 518 \text{ nm}$ , M.W = 364.911 g/mol, M.F = C<sub>23</sub>H<sub>25</sub>ClN<sub>2</sub>), 3. Crystal Violet (C.I. 42555,  $\lambda_{\text{max}} = 579 \text{ nm}$ , M.W = 407.99 g/mol, M.F = C<sub>25</sub>H<sub>30</sub>ClN<sub>3</sub>) (Note: M.W = Molecular weight; M.F = Molecular Formula). The dyes (adsorbates) and their chemical structures (Figure 1) utilized in this study are listed below.

### 2.4 Equipment

A UV/Vis spectrophotometer (Lambda 25) was used for measurement of dye concentration. The pH measurements were carried out using a Orion EA 940 expandable ion analyzer. An IHC- 3280 Orbital shaking incubator was used for all adsorption experiments. Centrifugation was done in a TC 650 S Multispin centrifuge.

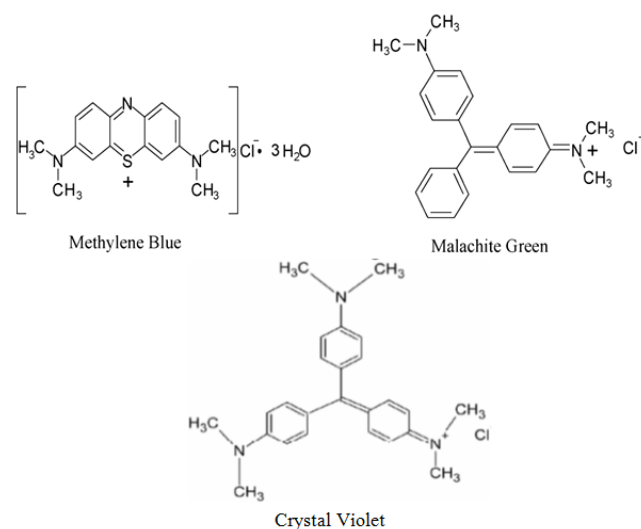


Figure 1. Chemical structures of dyes

## 2.5 Batch study

Adsorption studies were carried out by batch process for binary system (MG+MB, MG+CV, MB+CV) with initial concentration of 10 mg/L. The mixture (dye + adsorbent) was shaken well in an orbital shaking incubator at an agitation rate of 150 rpm at a temperature of 30°C. Batch kinetic experiments were carried out at constant pH of 8.0.

### 2.5.1 Effect of contact time

Kinetic study was conducted with the known dosage of adsorbent (bottom ash 0.05 g) for the 50 mL of dye solution. The samples were shaken at an agitation rate of 150 rpm. The samples were withdrawn at suitable time intervals. The sorbent solution mixtures were then centrifuged at 3600 rpm for 5 minutes and the supernatant was analyzed for the dye concentration. The difference between the initial and the final gives the amount of dye concentration removed.

### 2.5.2 Effect of adsorbent dosage

This study was carried out by shaking a series of bottles containing different amounts of adsorbents (0.01 to 0.1 g) in 50 mL of dye solution and kept in the shaker for constant duration (equilibrium time). The sorbent solution mixtures were then centrifuged at 3600 rpm for 5 minutes and the supernatant was analyzed for the dye concentration.

## 3. Results and Discussion

### 3.1 Characterization of adsorbent

The bottom ash used in the batch experiments was analyzed for various physical and chemical properties. The main physical properties of adsorbent were obtained as: point of zero charge of 6.2; specific gravity of 1.55 and loss of ignition of 8.15%. PZC of bottom ash shows that at pH less than 6.2 the surface of the bottom ash is predominated by positive charges while at pH greater than 6.2 the surface is predominated by negative charges (Nidheesh *et al.*, 2011). The solubility concentrations of some elements in water from bottom ash are: Cu ( $5.5 \times 10^{-3}$  mg/g), Na ( $5 \times 10^{-2}$  mg/g), K ( $1.625 \times 10^{-2}$  mg/g) and Cd ( $5 \times 10^{-4}$  mg/g). The chemical composition and FTIR spectrum of bottom ash used for this study has been given in our previous study (Nidheesh *et al.*, 2011). From the FTIR spectrum, it was found that bottom ash has peaks at 953, 1098, 1638, 3451  $\text{cm}^{-1}$  etc. The O–H stretching vibrations occurred within a broad range of frequencies indicating the presence of “free” hydroxyl groups and bonded O–H bands of carboxylic acids (Tan *et al.*, 2008). C–H stretching bands from aliphatic compounds occur in the range 3000–2850  $\text{cm}^{-1}$ . The main adsorption band for bottom ash is at 1000–1200  $\text{cm}^{-1}$  reflects the siloxane (–Si–O–Si–) group, and the bands at 786 and 714  $\text{cm}^{-1}$  represent SiO–H vibration (Lin *et al.*, 2007).

### 3.2 Effect of contact time

Dye uptake capacities were determined as a function of time to determine an optimum contact time for the adsorption of dye on bottom ash. The amount of dye adsorbed onto adsorbent was calculated by the difference between the content of the dye in the influent solution and that of the effluent solution, and it is expressed in percentage. The results are plotted in Figure 2. From the Figure 2 (a), it was observed that dye uptake in the binary system (MG+MB) was rapid for the first 4 minutes for MG and 5 minutes for MB and thereafter it proceeded at a slower rate and finally reached saturation. The uptakes of MG and MB were found

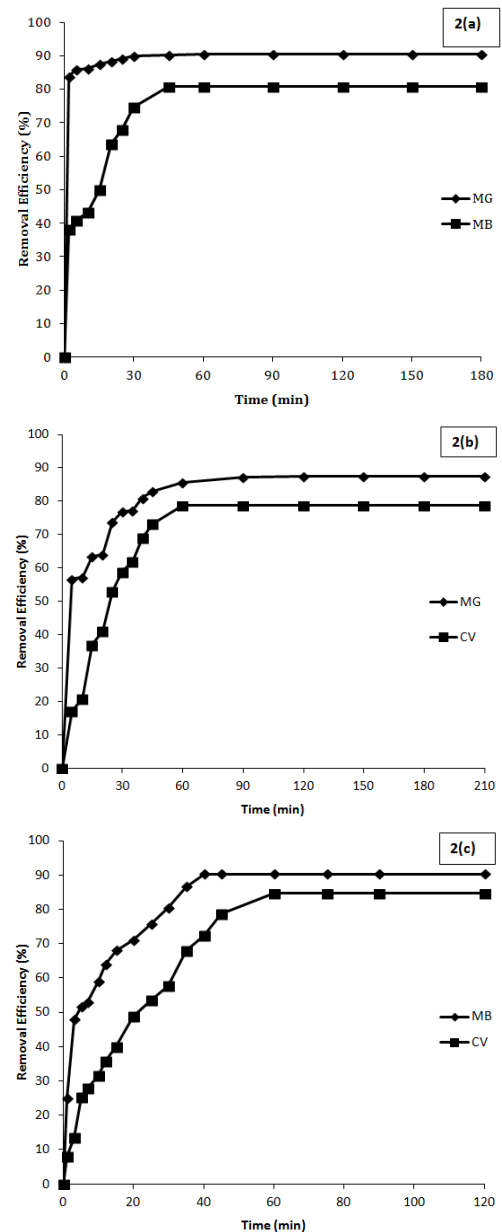


Figure 2. Effect of contact time for binary system (a) MG+MB (b) MG+CV (c) MB+CV

to be 90.34% and 80.7% at 30 minutes and 45 minutes respectively. From Figure 2(b), it was observed that dye uptake in the binary system (MG+CV) was rapid for the first 5 minutes for MG and 30 minutes for CV and thereafter it proceeded at a slower rate and finally reached saturation. The uptakes of MG and CV were found to be 87% and 78.7% at 90 minutes time interval. From Figure 2(c), it was observed that dye uptake in the binary system (MB+CV) was rapid for the first 7 minutes for MB and 10 minutes for CV and thereafter it proceeded at a slower rate and finally reached saturation. The uptakes of MB and CV were found to be 90.2% and 86% at 75 minutes and 45 minutes respectively.

### 3.3 Adsorption kinetics

In order to examine the controlling mechanism of adsorption processes such as mass transfer and chemical reaction, several kinetic models were used to test experimental data. The experimental data were also analyzed using the pseudo-first and pseudo-second-order adsorption kinetic models, and kinetic constants were calculated for concentration and adsorbent dose (Eren and Acar, 2006). The rate constant of adsorption is determined from the pseudo first-order equation given by Lagergren and Svenska (1989).

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \quad (1)$$

where  $q_e$  and  $q_t$  are the amounts of the dye adsorbed (mg/g) at equilibrium and at time  $t$  (min), respectively, and  $k_1$  is the rate constant adsorption ( $\text{min}^{-1}$ ). After integration and applying boundary conditions  $t = 0$  to  $t = t$  and  $q_t = 0$  to  $q_t = q_t$ , the integrated form of Eq. 1 becomes Eq. 2.

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t \quad (2)$$

The values of  $\log(q_e - q_t)$  were linearly correlated with  $t$ . The plot of  $\log(q_e - q_t)$  vs.  $t$  should give a linear relationship from which  $k_1$  and  $q_e$  can be determined from the slope and intercept of the plot, respectively.

The pseudo-second-order equation based on equilibrium adsorption (Ho and McKay, 1999) is expressed as:

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (3)$$

where  $k_2$  is the rate constant of pseudo-second-order adsorption (g/mg/min).

For the boundary conditions  $t = 0$  to  $t = t$  and  $q_t = 0$  to  $q_t = q_t$ , the integrated form of Eq. 3 becomes Eq. 4.

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 t \quad (4)$$

which is the integrated rate law for a pseudo-second-order reaction. Eq. 4 can be rearranged to obtain Eq. 5, which has a linear form

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

The plot of  $(t/q_t)$  and  $t$  of Eq. 5 should give a linear relationship from which  $q_e$  and  $k_2$  can be determined from the slope and intercept of the plot, respectively.

Experimental and theoretically calculated  $q_e$  values and coefficients related to kinetic plots are given in Table 1. Good fits were observed for all data where the adsorption reaction can be approximated with the second-order kinetic model. Based on the comparison between experimental and theoretically calculated  $q_e$  values, it was found that the pseudo-second-order model fitted better than pseudo-first-order model for all dye systems. It can also be observed from the Figure 3, that the second-order model predicted values are closer to experimental values than that of first-order predicted values. The rate of adsorption ( $k_1$ ) is equal for MB+CV system. It reveals that in MB+CV system, both basic dyes show same affinity towards fly ash. But the rate constant of pseudo-second-order adsorption ( $k_2$ ) is different in all the cases. This implies that all the binary systems have independent affinity towards fly ash.

Since neither the pseudo-first-order nor the second-order model can identify the diffusion mechanism, the kinetic results were analyzed by the intraparticle diffusion model. Rate of sorption is frequently used to analyze the nature of the 'rate-controlling step' and the use of the intraparticle diffusion model has been greatly explored in this regard and is represented by the following Weber and Morris equation (1963).

$$q_t = k_{id} t^{1/2} + C \quad (6)$$

Table 1 Kinetic coefficient for binary system

System	Dyes	Pseudo 1 <sup>st</sup> Order Coefficients			Pseudo 2 <sup>nd</sup> Order Coefficients			$q_e$ exp(mg/g)
		$q_e$ (mg/g)	$k_1$ (1/min)	$R^2$	$q_e$ (mg/g)	$k_2$ (g/mg/min)	$R^2$	
MG+MB	MG	1.327	0.021	0.867	9.346	1.041	1	9.145
	MB	6.516	0.041	0.971	8.475	0.028	0.998	8.440
MG+CV	MG	5.530	0.057	0.964	9.009	0.022	0.9995	8.700
	CV	10.313	0.058	0.955	8.749	0.007	0.9899	7.890
MB+CV	MB	6.186	0.069	0.936	9.488	0.023	0.9979	9.020
	CV	8.764	0.050	0.938	10.152	0.005	0.9854	8.460

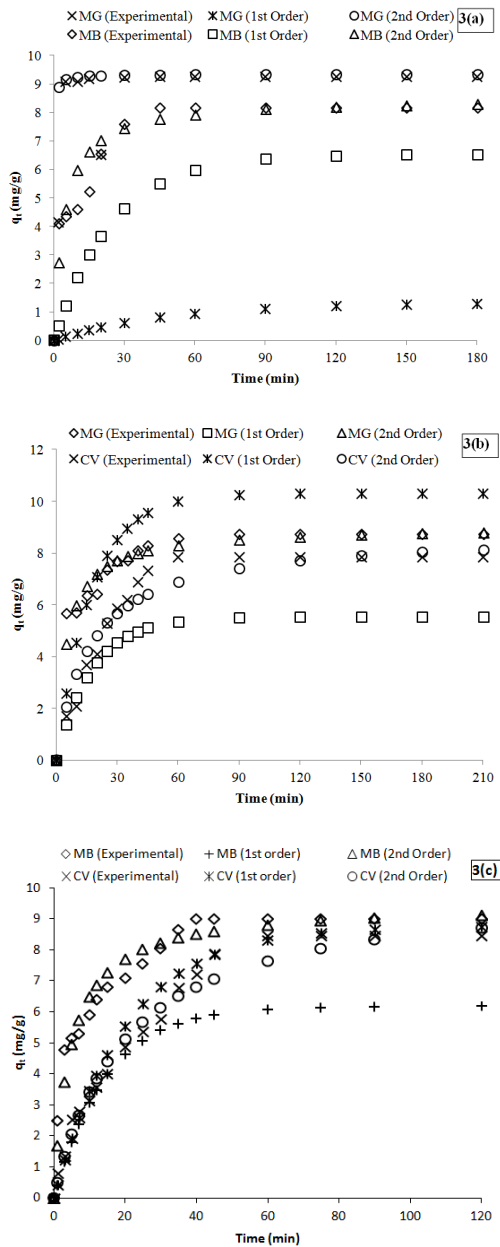


Figure 3. Pseudo 1<sup>st</sup> and 2<sup>nd</sup> order kinetics for binary system (a) MG+MB (b) MG+CV (c) MB+CV

where  $C$  is the intercept and  $k_{id}$  is the intraparticle diffusion rate constant ( $\text{mg/g min}^{0.5}$ ), which can be evaluated from the slope of the linear plot of  $q_t$  versus  $t^{1/2}$ . Figure 4 shows the plots of the intra particle diffusion model for adsorption of binary systems of basic dyes. The  $k_{id}$  values were obtained from the slope of the linear portions of the curve of different dye system and are shown in Table 2. According to this model, the plot of uptake,  $q_t$ , versus the square root of time ( $t^{1/2}$ ) should be linear if intraparticle diffusion is involved in the adsorption process and if these lines pass through the origin then intraparticle diffusion is the rate controlling step. When the plots do not pass through the origin, this is indicative of some degree of boundary layer control and this further

shows that the intraparticle diffusion is not the only rate-limiting step, but also other kinetic models may control the rate of adsorption, all of which may be operating simultaneously. The values of the intercept give an idea about the boundary layer thickness, such as the larger the intercept, the greater the boundary layer effect (Weber; Morris, 1963). As referred to Figure 4, there are two portions which suggest that two processes are involve, the surface sorption and intraparticle diffusion. Initial curve portion is attributed to boundary layer effect while the second linear portion is due to the intraparticle diffusion (Arivoli *et al.*, 2007). Deviation of the plot from the origin indicates that intraparticle diffusion is not the only rate-limiting mechanism for any of the binary systems.

### 3.4 Effect of adsorbent dosage

The effect of adsorbent dosage on the removal of dyes with different adsorbent doses for binary system, such as MG+MB, MG+CV and MB+CV is shown in Figure 5. The percentage removal of dyes increased with the increase in dose of adsorbent. Increase in adsorbent dose increased the percentage removal of dye, which is due to the increase in adsorbent surface area of the sorbent (Eren and Acar, 2006). It is evident from Figure 5 (a) that MG removal of 92% was observed at 0.9 g/L and 83.4 % for MB at 1.1 g/L. The MG removal of 92.3% was observed at 2.2 g/L and 81.8 % for CV at 1.4 g/L for the combination of MG and CV (Figure 5 (b)). From Figure 5 (c), it can be observed that the MB removal of 95.6% was observed at 1.6 g/L and 99.1 % for CV at 2g/L.

### 3.5 Binary adsorption isotherms

Multi-component equilibrium modelling has gone through many stages of development since the beginning of this century when none of the multi-component equilibrium studies dealt with competitive adsorption and a few only were concerned with the selectivity of adsorption processes (Chan *et al.*, 2012). Extended Langmuir (Noroozi *et al.*, 2008) and Freundlich (Baciocchi *et al.*, 2005) isotherms were used for analyzing the multi-component sorption process and their respective equations are given in Eqns. 7 and 8 respectively.

$$q_{e,i} = \frac{C_{e,i} b_i q_{m,i}}{1 + \sum b_i C_{e,i}} \quad (7)$$

Table 2. Intraparticle diffusion constants

Systems	Dyes	$K_{i,1}$	$K_{i,2}$	$C_1$	$C_2$
Binary	MG	0.123	-	8.264	0344
System 1	MB	0.8942	-	2.3453	8.161
Binary	MG	0.6141	0.0019	0.655	8.7146
System 2	CV	1.342	-	1.636	7.87
Binary	MB	1.0724	-	2.4008	9.02
System 3	CV	1.215	-	0.5453	8.46

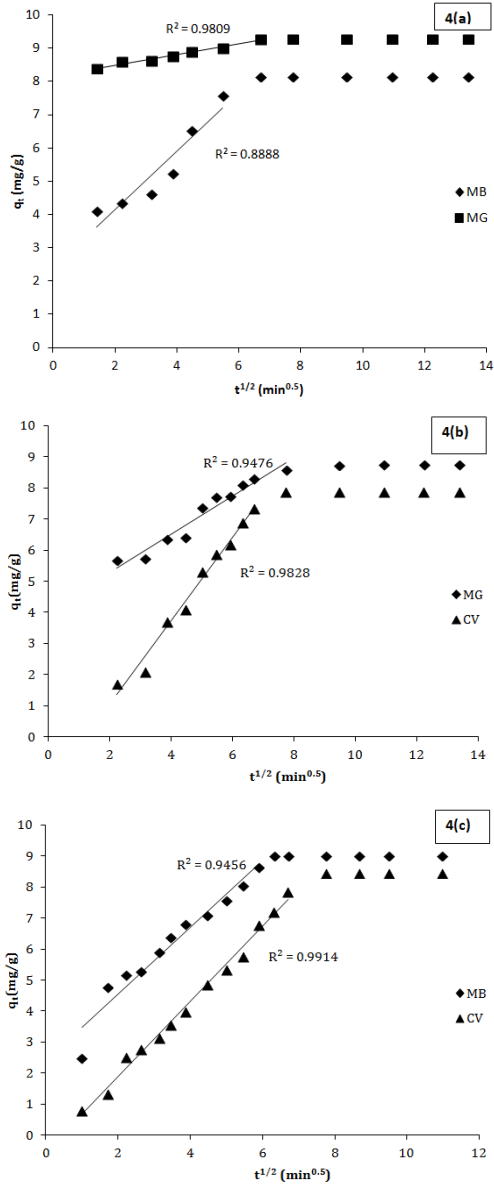


Figure 4. Intraparticle diffusion model for binary system (a) MG+MB (b) MG+CV (c) MB+CV

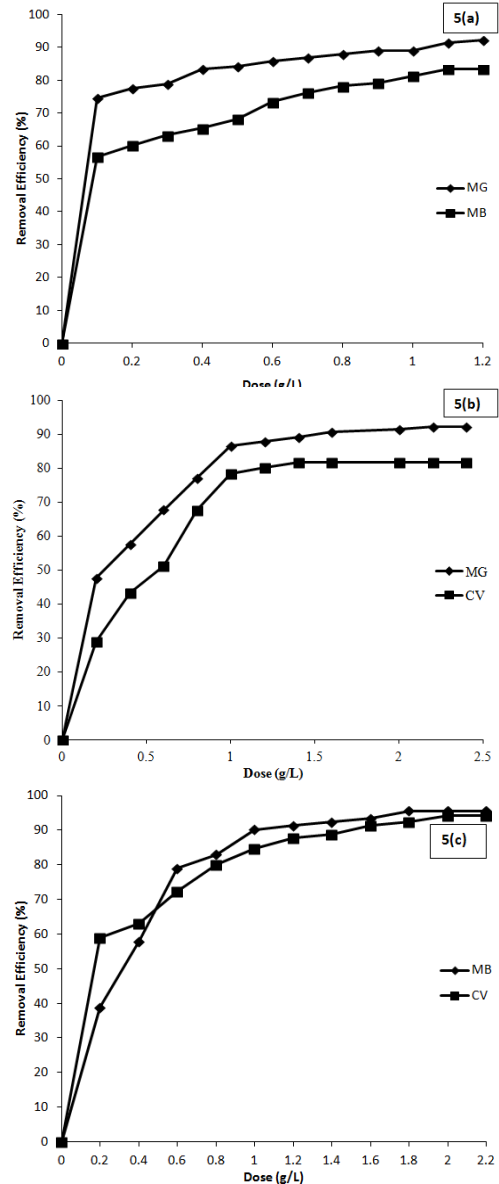


Figure 5. Effect of dose for binary system (a) MG+MB (b) MG+CV (c) MB+CV

$$q_{e,i} = K_{f,i} C_{e,i} (C_{e,i} + \sum_{j=1, j \neq i}^N \alpha_{i,j} C_{e,j})^{1/n_i - 1} \quad (8)$$

Where  $\alpha_{i,j} = \frac{b_j}{b_i}$  (9)

where  $q_{m,i}$  and  $b_i$  are the Langmuir isotherm constants obtained from the single system,  $K_{f,i}$  and  $n_i$  are the Freundlich isotherm constants obtained from the single system,  $q_{e,i}$  is the amount of component  $i$  adsorbed after equilibrium and  $C_{e,i}$  is the equilibrium concentration of component  $i$ .

The suitability of extended Langmuir and Freundlich isotherms for the experimental data is checked using sum of squares of relative error (SSRE). SSRE is given as (Noroozi *et al.*, 2008):

$$SSRE = \sum \left( \frac{(q_{e \text{ expt}} - q_{e \text{ model}})}{q_{e \text{ expt}}} \right)^2 \quad (10)$$

where  $q_{e \text{ expt}}$  is the uptake of dye calculated from experiment and  $q_{e \text{ model}}$  is the uptake of dye predicted by competitive isotherm models.

From our previous study (Gandhimathi *et al.*, 2012), single MB and CV sorption capacities of bottom ash from Freundlich isotherms were found as 10.86 and 12.1 mg/g with  $n$  values of 1.175 and 2.89 respectively. Similarly, Langmuir isotherm predicted maximum equilibrium uptake as 151.52 and 13.6 mg/g respectively, for MB and CV. MG sorption by bottom ash was not favored by both isotherms. Therefore, competitive isotherms of MB and CV were considered for

Table 3 Isotherm constants for MB+CV binary system

Dyes	Extended Langmuir $q_e$ (mg/g)	Isotherm SSRE	Extended Freundlich $q_e$ (mg/g)	Isotherm SSRE
MB	4.1	7.23	5.27	0.99
CV	3.06	0.48	4.04	0.25

the present study.

The calculated  $q_e$  values from both competitive isotherms along with SSRE values are given in Table 3. From this table, it can be observed that the adsorption capacity of bottom ash in binary system is less than that of the single system for each of the dyes. The value of SSRE is very much less in most of the cases, indicating that the competitive sorption of dyes on bottom ash is well predicted by both isotherms. The comparison of model predicted and experimental values is shown in Figure 6. From Figure 6, it can be observed that the model predicted values are very close to the experimental values.

#### 4. Conclusions

Bottom ash, a thermal power plant waste has the ability to remove basic dyes (MG, MB and CV) from binary solutions. The optimum contact time and optimum dose for MG were found to be 30 min and 9 g/L with the removal efficiency 92%, and for MB 45 min and 1.1 g/L with 83.4% removal. For the binary system (MG+CV) the optimum contact time and the optimum dose for MG were found to be 90 min and 2.2 g/L with the removal efficiency 92.3%, and for CV the corresponding values were 90 min and 1.4 g/L with 81.8% removal, and for the binary system (MG+CV) the values were for MG were 45 min and 1.6 g/L with the removal efficiency 95.6% and for CV these values were 60 min and 2 g/L with 94.1% removal. In the kinetic study, pseudo 1<sup>st</sup> and 2<sup>nd</sup>-order models were fitted for all dye systems. Based on the experimental sorption capacity values compared with model

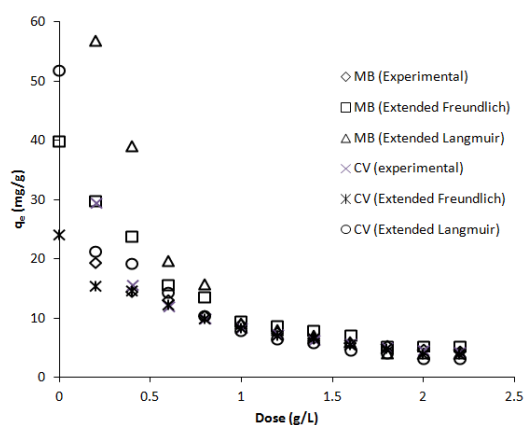


Figure 6. Comparison of model predicted and experimental values for MB+CV binary system

values it may be concluded that pseudo-second-order model fitted better than pseudo-first-order model for all system of MG, MB and CV. From the effect of adsorbent dose, it can be concluded that increasing the adsorbent concentrations increased the removal efficiency, but adsorption capacity (i.e. adsorption amount per unit mass) decreased. Langmuir and Freundlich isotherm models were fitted for all systems of dyes; Freundlich isotherm model was found to be the best fit for all systems.

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