Adsorption Isotherm Models and Kinetics for Chlorpyrifos Adsorption from Aqueous Solutions onto Bottom Ash using Batch Experiments

Priya Kumari¹, Masood Alam²

Department of Applied Sciences & Humanities, Faculty of Engineering and Technology, Jamia Millia Islamia, New Delhi 110025, India

Abstract: The ability of activated bottom ash (ABA) to adsorb chlorpyrifos from aqueous solution has been studied through batch experiment. The ABA was characterized by FTIR (Fourier Transform Infrared Spectroscopy) and SEM (Scanning Electron Microscopy). The batch experiment has been performed by varying Contact time, pH, Adsorbate concentration and Adsorbent dose. The adsorption process is found to be pH-dependent, maximum adsorption takes place at pH 2. The adsorption process was relatively moderate, and equilibrium achieved after 120 min of contact time. The adsorption equilibrium data was, then, fitted to Langmuir, Freundlich and Temkin isotherm models one by one to evaluate the mode of adsorption. Experimental results showed that, Langmuir isotherm describes this adsorption process the best. The kinetics of adsorption was further analysed by applying the pseudo-first-order, the pseudo-second-order and the Weber-Morris diffusion model to adsorption data. The kinetic data of this adsorption obeyed pseudo-second-order rate equation.

Keywords: Adsorption, bottom ash, isotherm, kinetics

1. Introduction

More than two third of Earth surface is covered with water which is one of the vital resource for sustaining life on Earth. Despite of that, millions on planet are still struggling to get potable water. Scarcity of potable or drinkable water is becoming bigger problem day by day. In the bygone decades, the demand of clean water has increased by leaps and bounds due to Industrial growth as well as increasing population [1]. Consequently, water pollution effects human health and environment alike. Unfortunately, water pollution has now become such a major concern that Academicians and Scientists are coming together to brainstorm with Govt. Authorities to launch well planned Government Schemes in order to tackle this "National Issue". Countless biological, organic and inorganic chemicals have been listed as water pollutants, their effects ranging from being slightly toxic to hazardously fatal to human health [2]. Exponentially growing population, industrial wastes, various agricultural pesticides are few of the several sources of Water Pollution. The ever growing industries and their industrial waste are continuously polluting the soil, water as well as air around us. The various industrial bi-products which are being continuously discharged into the rivers and other water bodies have various pollutants and thus, entering our ecosystem. It has significant and toxic impact over the lives of humans, plants and animals alike. In places like local villages or small / medium cities, Ground Water serves as the primary water source for daily needs. But, increasing use of pesticides and fertilizers in agricultural activities is polluting the ground water quality as well.

The insecticides form a very strong class of water pollutants owing to their carcinogenic nature. Moreover, they are sometimes, non-biodegradable as well, making them further more hazardous [3]. There is enough evidence showing that many of the water resources in India have been polluted with several pesticides. Chlorpyrifos is a typical example of a commonly used agricultural insecticide which reaches humans from food residues and our water streams through various irrigation activities. Though it has its uses as an important organophosphate pesticide, if mixed to water streams, it can be very hazardous [4]. It is very difficult to remove it using conventional wastewater treatment processes and resultantly that makes its separation from the water streams very important and necessary.

The wide range of pesticides / insecticides makes it hard for the researchers to establish a single universal method for removal of insecticides. Hence, many methods are individually or in conjunction are applied for the waste removal from water which includes chemical oxidation with ozone [5], photo degradation [6], combined ozone and UV irradiation [7], Fenton degradation [8], biological degradation [9], coagulation [10], and adsorption [11-13].

The adsorption technique is a very effective and widely accepted method [14] for removing both organic as well as inorganic micro - pollutants from water. Activated carbon is one of the most commonly available adsorbent from industrial point of view [15]. However, despite being very effective in wastewater treatment, carbon adsorption is still a very expensive procedure and has its drawbacks. Preparation and regeneration of activated carbon is a very tedious procedure and the operating cost is also very high [16]. Hence despite being effective, its use in the field is often restricted on economic grounds.

Fortunately, in recent times, a lot of work is being done towards exploring the potential of industrial by-products for their use as adsorbents [17] such as Coal Fly Ash [18], Bagasse Fly Ash [19], Banana Pith [20], Bagasse Pith [21], Orange peel [22], chitosan [23], coir pith [24], rice husk [25], [26] and many more. However, study shows that some of the

adsorbents used have low capacity as compared to activated carbon and are also not versatile enough to adsorb a variety of pesticides.

Bottom Ash is one such industrial waste which is very cheap and easily available in abundance from Thermal Power Plants. In the present work, attempts are made to use bottom ash, a non-combustible residue and a waste material produced during combustion in a furnace in any thermal power station, as a useful and efficient low cost adsorbent. It was collected from BTPS (Badarpur Thermal Power Station), New Delhi, India. The present research article shows as how efficiently Bottom Ash can be used as an adsorbent in the removal of chlorpyrifos (the pollutant) from the aqueous solution.

2. Experimental Methodology

The bottom ash, when initially procured from the plant, was in the form of small, spherical, greyish black, uneven sized particles. To begin with, Bottom Ash was sieved with mesh sized BSS-72 and then washed with deionised water. Later, it was thoroughly dried in an oven at 100 °C for 5 hours. Further, the dried and cleaned (purified) Bottom Ash was chemically treated with hydrogen peroxide solution (about 30% w/v). For activation of bottom ash, it was heated at 500° C for 20 min in a muffle furnace and then stored in desiccator for further use. Finally, chemically treated and activated Bottom Ash was used as an adsorbent for removing chlorpyrifos from the aqueous solution.

Chlorpyrifos was procured from Sigma-Aldrich and 50 ppm stock solution of pesticide was prepared in hexane. All reagents used were of HPLC grade. Further, standard solutions of pesticide with lower concentration were prepared by successive dilutions.

During the course of adsorption, pH testing was done by using pH meter. The characterization of bottom ash was done, by recording IR spectrum using Tensor 37 FTIR spectrophotometer and SEM was performed on FEI Nova NanoSEM 450 electron microscope. The adsorption experiment was performed on an Electromagnetic stirrer, using beakers (volume: 100 ml).

3. Results and Discussion

3.1 Characterization of Bottom ash

FTIR was used for the qualitative analysis of ABA, knowing that the strength of adsorption depends upon concentration. The FTIR spectrum of bottom ash, before chemical treatment, after chemical treatment and after adsorption is given in Figure 1. The peaks present at 2240 cm⁻¹ and 2177 cm⁻¹ reveals the presence of C=N and -C=C- stretching respectively. Other significant peaks are shown as follows, at 1738 cm⁻¹ (C=O stretching), 1563 cm⁻¹ and 1366 cm⁻¹ (N–O stretch), 1439 cm⁻¹ (aromatic C=C stretch), 1052 cm⁻¹ (C–O stretch), 884 cm⁻¹, 782 cm⁻¹, 638 cm⁻¹ (=C–H bending). The complex nature of the adsorbent can be clearly deduced from the various absorption peaks of the functional groups in the

spectrum. Comparing FTIR spectrum before and after chemical treatment and adsorption, it is clear that few peaks at 2240 cm⁻¹ (C=N stretch), 2177 cm⁻¹ (-C=C- stretch), 1738 cm⁻¹ (C=O stretch), 884 cm⁻¹ and 638 cm⁻¹ (=C-H bending) have shifted and new peaks at 1737 cm⁻¹ (C=O stretching), 2354 cm⁻¹ (N-H stretch) emerged, after adsorption. These changes in spectrum clearly indicate the ongoing interactions amongst the available reactive groups present at the surface of bottom ash and chlorpyrifos.



Figure 1: FTIR spectrum of bottom ash



Figure 2(a): SEM image of bottom ash before chemical treatment



Figure 2(b): SEM image of bottom ash after chemical treatment

SEM micrographs were also obtained for bottom ash at three different stages, (a) Before chemical treatment, (b) After chemical treatment and (c) After adsorption of chlorpyrifos. There is a visible change in surface morphology of bottom ash at each stage. After chemical treatment, surface of bottom

ash becomes more distorted as shown in Fig. 2(b), providing more surface area for the adsorption process in comparison to Fig. 2(a) i.e., before chemical treatment. Further, Fig. 2(c) shows continuous structure due to the adsorption of chlorpyrifos on the surface of bottom ash.



Figure 2(c): SEM image of bottom ash after adsorption

3.2 Adsorption study

3.2.1 Effect of contact time

The removal efficiency of ABA for chlorpyrifos from aqueous solution with variable contact time is shown in Figure 3(a). To attain maximum uptake equilibrium time, 1 g of ABA was used at pH 2 for 5 ppm solution of chlorpyrifos for contact time of 30-150 min and increasing the contact time by 30 min at each step. The percentage adsorption of the pesticide increases as the contact time increases and attains the equilibrium at 120 min of contact time. The percentage adsorption increases from 78% to 96% with the increase in contact time.



Figure 3(a): Change in adsorption capacity with contact time

3.2.2 Effect of adsorbate concentration

The percentage removal of chlorpyrifos from solution using bottom ash with variable concentrations of pesticide is shown in Figure 3(b). The concentration of pesticide was varied from 1 ppm to 5 ppm, at fixed pH (2), contact time (120 min), dose (1 g) and temperature (28° C). The adsorption capacity decreases from 97% to 78% with the increase in concentration, as the number of vacant sites on adsorbent surface become less with the increase in pesticide concentration.



Figure 3(b): Change in adsorption capacity with adsorbate concentration

3.2.3 Effect of pH

The percentage removal of chlorpyrifos at variable pH values is shown in Figure 3(c). The parameters other than pH, namely contact time (120 min), adsorbate concentration (5 ppm), dose (1 g) were kept constant and pH values were varied from 2 to 10. The removal capacity dropped from 97% to 51% with the pH increase from 2 to 10. Deprotonation of functional groups at the surface of bottom ash, produces negative charge, which causes electrostatic repulsion between bottom ash and chlopyrifos.



Figure 3(c): Change in adsorption capacity with pH

3.2.4 Effect of adsorbent dose

The influence of variation in ABA dose was also studied by keeping the concentration of the pesticide constant as shown in figure 3(d). The adsorbent doses were varied from 0.5 to 4 g at fixed pH (2), contact time (120 min) and adsorbate concentration (5 ppm). As the number of available sites for adsorption increases with the increase in adsorption dose, percentage adsorption increased from 78% to 99% with the increase in adsorbent dose.



dose

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3.3 Adsorption isotherm

To propose an efficient pesticide removal experiment, data obtained from adsorption experiment using four variables namely contact time, adsorbate concentration, pH and adsorbent dose were applied to three different isotherm models. The adsorption isotherm depicts that the interactions takes place between adsorbate and adsorbent species during adsorption process and gives us a point where a state of equilibrium is achieved. To evaluate the mechanistic parameters, experimental equilibrium data of chlorpyrifos applied to bottom ash was analysed using Langmuir, Freundlich and Temkin isotherm model. The correlation coefficient (R^2) was calculated for each of the isotherm model to evaluate the adsorption isotherm.

The Langmuir isotherm proposes formation of saturated monolayer of adsorbate (in this case chlorpyrifos) on the surface of adsorbent, at the available sites [27]. Linear form of the Langmuir isotherm model can be presented as given in equation (1):

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \left(\frac{1}{q_m}\right)C_e \quad \dots \quad (1)$$

where q_m (mg g⁻¹) represents Langmuir constant (adsorption capacity) and b (L mg⁻¹) is the rate of adsorption, q_e (mg g⁻¹) is the amount of chlorpyrifos adsorbed at the equilibrium and C_e (mg L⁻¹) is the equilibrium concentration.

The values of Langmuir constants were found from the intercept and slope of the linear curve C_e/q_e versus C_e as given in figure 4(a). The values of Langmuir constants q_m , b with R^2 are given in table 1. The separation factor (R_L) is a dimensionless constant which can express the characteristics of Langmuir isotherm. This constant is defined as:

$$R_L = \frac{1}{1+bC_o} \qquad \dots \dots (2)$$

Where C_o is the initial pesticide concentration (ppm) and b is the Langmuir constant. The value of R_L obtained gives us an idea about the type of Langmuir isotherm: linear ($R_L = 1$), irreversible ($R_L = 0$), favourable ($0 < R_L < 1$) or unfavourable ($R_L > 1$).

In this case the values of R_L were found to be 0.4-0.8 showing the favourable isotherm. It concludes that Langmuir isotherm is well fitted to the adsorption of chlorpyrifos on bottom ash under the aforesaid maintained conditions.

The linear form of Freundlich isotherm equation as applied to present adsorption system can be given in equation 3.

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad \dots \dots (3)$$

where n and K_f are the Freundlich equilibrium constants related to intensity of adsorption and adsorption capacity, respectively. The linear plot of log q_e versus log C_e in figure 4(b) gives the values of Freundlich constants. The corresponding K_f and n values are reported in table 1 along with R^2 (correlation coefficient) value. The values of n ranging from 1 - 10 represents a favourable adsorption, hence in this case Feundlich isotherm is not favourable.

 Table 1: Isotherm model parameters and R² value for adsorption of chlorpyrifos onto ABA

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Pesticide	Isotherm Model	Constants							
Chlorpyrifos	Langmuir	$q_m (mg/g)$	b (L/mg)	\mathbb{R}^2					
	Model	0.044	0.236	0.975					
	Freundlich	n	K_{f} (mg/g)	\mathbb{R}^2					
	Model	0.809	0.173	0.920					
	Temkin	$A_T (L/g)$	В	\mathbb{R}^2					
	Model	3.966	0.123	0.830					

The Temkin adsorption isotherm model (equation 4) was applied to examine the influence of indirect interactions amongst the particles of adsorbate. Temkin model suggests that as the surface coverage increases due to the interactions of adsorbate and adsorbent, the heat of adsorption of all the molecules in the layer deceases [28]. The linear form of the Temkin isotherm equation can be given by equation 5.

$$q_e = \left(\frac{RT}{b}\right) Ln A C_e \qquad \dots \dots (4)$$

$$q_e = B \ln A_T + B \ln C_e \quad \dots \dots (5)$$

Where A_T (L g⁻¹) is the equilibrium binding constant, b (J/mol) is the heat of adsorption related constant, R is the gas constant (8.314 J/mol/K), and T is absolute temperature (K). The linear plot q_e versus ln C_e in figure 4(c) gives the values of A_T and B from intercept and slope, respectively. The values of Temkin constants are given in table 1 along with the correlation coefficient. The plot obtained is not a straight line suggesting non-uniform distribution of binding energy.

Thus, focusing on table 1 it is clear that Langmuir isotherm $(R^2 = 0.97)$ fits well to the adsorption process of chlorpyrifos onto bottom ash. Whereas the low values for other isotherm model, Freundlich ($R^2 = 0.92$) and Temkin ($R^2 = 0.83$) shows poor agreement with the adsorption data.



Figure 4(a): Langmuir isotherm for chlorpyrifos adsorption on bottom ash



Figure 4(b): Freundlich isotherm for chlorpyrifos adsorption on bottom ash

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Figure 4(c): Temkin isotherm for chlorpyrifos adsorption on bottom ash

3.4 Adsorption kinetics

In order to investigate the adsorption process of chlorpyrifos on bottom ash, three kinetic models were used, including pseudo-first-order, pseudo-second-order and intra-particle diffusion model. The procedure used for equilibrium tests was used for kinetic tests as well. The aqueous solution of pesticide with fixed concentration, dose and pH was taken and the concentration was measured at the time interval of 30 min. The amount of chlorpyrifos adsorbed; q_t (mg g⁻¹), at the surface of bottom ash at time; t, was calculated using equation 6.

The pseudo-first-order equation proposed by Langergren and Svenska as given by equation 7 was used to determine the rate constant of adsorption.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \dots (7)$$

where q_e and q_t (mg g⁻¹) are the amounts of pesticide adsorbed at equilibrium and time, t (h) and k_1 (h⁻¹) is the adsorption rate constant. To calculate the values of k_1 and q_e by using pseudo-first-order kinetics plot of ln ($q_e - q_t$) versus t for the initial 5ppm concentration of the pesticide was drawn, which has been presented in figure 5(a). The values of kinetic parameters from equation 7 along with correlation coefficient are given in table 2.

Table 2: Rate constants of Pseudo-first-order and pseudo-second-order for adsorption of chlorpyrifos onto ABA

pesticide	q _e expt	Pseudo-first-order			Pseudo-second-order		
pesticide	(mg/g)	$q_e(mg/g)$	k ₁	\mathbf{R}^2	$q_e(mg/g)$	k ₂	\mathbf{R}^2
Chlor- pyrifos	0.048	0.014	0.016	0.990	0.048	3.178	1

The pseudo-second-order kinetics for equilibrium adsorption can be given by equation 8.

$$\frac{t}{q_t} = \frac{1}{k_2 \cdot q_e^2} + \frac{1}{q_e}t \quad \dots (8)$$

where k_2 (g mg⁻¹ h⁻¹) is the rate constant of second-order adsorption. To calculate the values of q_e and k_2 by using pseudo-second-order kinetics, plot of t versus t/q_t are presented in figure 5(b). The values of kinetic parameters from equation 8 are given in table 2. It is clear that the correlation coefficient for pseudo-second-order is greater than that of pseudo-first-order and the experimental q_e value agrees with the observed q_e value of pseudo-second-order equation. This adsorption process therefore, follows pseudosecond-order kinetics.



Figure 5(a): Pseudo-first-order kinetics of chlorpyrifos adsorption on bottom ash



Figure 5(b): Pseudo-second-order kinetics of chlorpyrifos adsorption on bottom ash

Weber and Morris proposed an intra-particle diffusion model to identify the diffusion mechanism of adsorption. According to this model adsorbate uptake varies proportional to $t^{1/2}$ and not to the contact time t [29]. Equation 9 which is given below represents the linear form of the intra-particle diffusion model.

$$q_t = K_{id} t^{1/2} + I \quad \dots \dots \quad (9)$$

where K_{id} is the intra-particle diffusion rate constant and I is the thickness of the boundary layer. The plot of q_t versus $t^{1/2}$ shown in figure 5(c), gives the value of K_{id} and I from, slope and intercept, respectively. Figure 5(c), shows multistage linearity involving moderate diffusion rate at the initial stage, followed by rapid diffusion, indicating that the intra-particle diffusion is the rate controlling adsorption process [30], [31].



Figure 5(c): Weber and Moris plot of chlorpyrifos adsorption on bottom ash

4. Conclusion

The present study shows that the bottom ash can be used as an adsorbent for removing chlorpyrifos from aqueous solutions. SEM images verified that the surface of bottom ash after chemical treatment becomes more distorted and results

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in higher number of vacancies for adsorption. In the batch experiments, equilibrium was achieved within 120 min. The percent removal of pesticide in 120 min with variable dose increased from 78% to 99%. The maximum removal of pesticide occurs at pH 2. On varying the initial concentration from 1-5 mg L^{-1} the percent removal deceases from 97% to 78% as the number of vacant sites for adsorption become less. Out of three isotherm models, Langmuir isotherm model $(R^2 = 0.97)$ fitted best to the adsorption process, as compared to Freundlich isotherm ($R^2 = 0.92$) and Temkin isotherm (R^2 = 0.83) model. Langmuir model suggests that the formation of a monolayer of chlorpyrifos takes place at the surface of bottom ash. Pseudo-second-order activated kinetics explained the adsorption process better than pseudo-firstorder. It can, therefore, be concluded that, the activated bottom ash which is available in abundance can be used as an efficient and economical adsorbent for the removal of pesticides from aqueous solution.

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Author Profile



Prof. Masood Alam is a renowned academician and researcher at the Department of Applied Sciences and Humanities, Faculty of Engineering and Technology, Jamia Millia Islamia (Central University), New Delhi, Jaci Markov, New Delhi, and Ph.D. degrees from

India. He received his M.Sc., M.Phil. and Ph.D. degrees from Department of Chemistry, Aligarh Muslim University, Aligarh, India in 1979, 1981 and 1983 respectively. His research areas include Analytical Chemistry and Environmental studies. He has published more than 50 papers in National and International journals. Prof. Alam is also a life member of Indian Society for Technical Education. He has held various administrative positions in Jamia Millia Islamia, New Delhi, India including Proctor, Chief Proctor and Security advisor to VC.



Ms. Priya Kumari is a research scholar at the Department of Applied Sciences and Humanities, Faculty of Engineering and Technology, Jamia Millia Islamia (Central University), New Delhi, India. She

obtained her M.Sc. and M.Phil. in Chemistry from Dayalbagh Educational Institute, Agra, India. She is pursuing her Ph.D. under the supervision of Prof. Masood Alam form October, 2015. Her areas of research are Air Quality Assessment and Water Treatment. Present work represents synthesis of new economical and ecofriendly adsorbents for environmental benefit.

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