



Batch and Column Operations for the Removal of Fluoride from Aqueous Solution Using Bottom Ash

S. T. Ramesh, R. Gandhimathi, P. V. Nidheesh and M. Taywade

Department of Civil Engineering, National Institute of Technology, Tiruchirappalli, Tamil Nadu, India.

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Millions of people rely on drinking water that contains excess fluoride. In fluoride endemic areas, especially small communities with staggered habitat, defluoridation of potable water supply is still a problem. In this study, adsorption potential of bottom ash was investigated for defluoridation of drinking water using batch and continuous fixed bed column studies. Batch sorptive defluoridation conducted under experimental conditions such as pH, temperature, particle size, agitation time and dosage. Adsorption isotherms have been modeled by Langmuir and Freundlich isotherms. The fluoride sorption capacity at a breakthrough point for bottom ash was greatly influenced by bed depth. The results were then analyzed using the Bed Depth Service Time (BDST) Model, and various performance parameters like Adsorption Capacity, Critical Bed Depth, and Adsorption Rate Constant were evaluated. With the observed data the Thomas model and the Yoon Nelson model fitted well and the constants were found.

Key words: Adsorption; Bottom ash; Batch study; Fixed-bed column study; Kinetics; Isotherms.

1. Introduction

Fluoride is known to be a natural contaminant for ground water resources globally. Fluorine, a fairly common element of the earth crust, is present in the form of fluorides in a number of minerals and in many rocks (Chidambaram et al. 2001). Recalling the basics of inorganic chemistry we should keep in mind that among the four best known halogens (chlorine, bromine, iodine, and fluorine) namely fluorine is the most chemically active, being a very strong oxidant (Veressinina et al. 2001). "The ingestion of excess fluoride can cause fluorosis which affects the teeth and bones. Moderate amounts lead to dental effects, but long-term ingestion of large amounts can lead to potentially severe skeletal problems. Paradoxically, low levels of fluoride intake help to prevent dental caries" (Sehn 2008). Research of several researchers during the last 5–6 years has proved that life-long impact and accumulation of fluorides cause not only human skeletal and teeth damage, but also changes in the DNA-structure, paralysis of volition, cancer, etc (Veressinina et al. 2001). Mainly two factors are responsible for contamination of ground water with fluoride –

geological and anthropogenic. Although both geological and manmade sources contribute to the occurrence of fluoride in water, the major contribution comes from geological resources (Nath and Dutta 2010). In the 3rd edition of the World Health Organization (WHO) guidelines on drinking water, it maintains its guideline on the appropriate fluoride concentration at 1.5 mg/L (WHO 2008). "It is estimated that around 260 million people worldwide (in 30 countries) are drinking water with Fluoride content more than 1.0 mg/l. In India alone, endemic Fluorosis is thought to affect around one million people and is a major problem in 17 of the 25 states, especially Rajasthan, Andhra Pradesh, Tamil Nadu, Gujarat and Uttar Pradesh" (Vardhan and Karthikeyan 2011). So Fluoride removal from water is an important mission of Environmental Engineers.

Adsorption is one of the significant techniques in which fluoride adsorbed onto a membrane, or a fixed bed, is packed with resin or other mineral particles (Alagumuthu et al. 2010). They are also among the most effective and easy to operate and thus show wider applicability (Gupta et

al. 2009). Even though activated carbon, a common adsorbent used in water and wastewater treatment, showed advantages, the main drawback of the activated 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). Many natural and low cost materials such as Pumice (Malakootian et al. 2011), Rice husk (Vardhan and Karthikeyan 2011), *Moringa Oleifera* seeds (Vardhan and Karthikeyan 2011), Bermuda grass (Alagumuthu et al. 2010), cashew nut shell carbon (Alagumuthu and Rajan 2010) etc have been used as adsorbents for fluoride removal from drinking water.

The present paper is an attempt to explore a possibility to utilize a waste material adsorbent, 'bottom ash' to remove fluoride from aqueous solution. The 'bottom ash' is a waste material obtained by thermal power generation plants after combusting solid fuels. It is an undesired collected material, which is transported and dumped near the surrounding land. Its disposal has always been a matter of concern to the station authorities, as the dumped ash is considered highly unsuitable for the agricultural utilization (Gupta et al. 2004). Already it is proved that bottom ash is very efficient adsorbent for removing malachite green (Gupta et al. 2004), 2-Aminophenol (Gupta et al. 2006), Basic fuchsin (Gupta et al. 2008), Chrysoidine Y (Mittal et al. 2010) and Crystal Violet (Nidheesh et al. 2011). The paper presents adsorption kinetics and equilibrium uptake of fluoride over bottom ash. The effect of contact time, pH, temperature, particle size, bottom ash dosage and speed on fluoride removal were studied. Column operations were carried out to develop an industrial application of this research. Bed Depth Service Time (BDST), the Thomas and Yoon Nelson models were used to analyze the column experimental data.

2. Materials and Methods

Adsorbent

The thermal power plant waste material 'Bottom ash' was utilized as adsorbent in the study. The adsorbent was obtained from the Neyveli Lignite Corporation, Neyveli, Tamil Nadu. Bottom ash of size 300 μm was collected for batch studies column studies.

Equipment

The pH measurements were obtained using an Orion EA 940 expandable ion analyzer. An IHC 3280 Orbital shaking incubator was used for all adsorption experiments.

Preparation of Stock Solution

The fluoride stock solution of 1000 mg/l was prepared by diluting 2.21 g of sodium fluoride salt in one liter of distilled water.

Preparation of TISAB-II

Total ionic strength adjustment buffer solution prepared with NaCl (58 g) salt diluted into 500 ml of distilled water and 57 ml of acetic acid was added. The solution cooled in a water bath and after cooling pH of that solution was maintained at 5-5.5 with adding 5 M NaOH solution. The solution was diluted up to 1000 ml with distilled water in a standard flask.

Batch Studies

In order to study the effect of different controlling parameters like pH, contact time, dosage etc. on defluoridation capacity of bottom ash, adsorption studies are carried out by a batch process. Batch equilibrium experiments were conducted using a total sample volume of 100 ml for each adsorption run. The samples were agitated in a reciprocating shaker to reach equilibrium. Suspensions were then centrifuged at 2500 rpm for 2 minutes and then supernatant collected. The samples were analyzed for a fluoride concentration. All the experiments were carried out under natural conditions.

Fixed-Bed Column Studies

The sorption studies were carried out in a glass column of 2.5 cm diameter and bed height of 20 cm. Packed bed experiments were carried out at 33°C. The column experiments were conducted with 10 mg/l fluoride solutions at bed depths of 10 cm, 15 cm, and 20 cm, respectively, at a constant flow rate of 10 ml/min.

3. Results and Discussions

Effect of Contact Time and Temperature

Fluoride ion uptake capacities were determined as a function of time to determine an optimum contact time for the adsorption of fluoride ion on bottom ash. Fig. 1 shows time course of the adsorption of fluoride onto bottom ash at 293 K, 303 K and 313 K. It has been observed that the fluoride adsorption rate of bottom ash is high at the beginning and then decreases slowly till saturation levels are completely reached at the equilibration point. But the equilibration time decreases with the temperature without much increase in fluoride ion uptake. According to the results as shown in Fig. 1, it was observed that the time necessary to reach the equilibrium was 165 min at 293K. When the temperature varied from 303K to 313K, the equilibrium time decreased from 135 to 120min. Results indicated that the time to reach equilibrium was slightly affected by the temperature of fluoride solution.

Effect of Adsorbent dosage

The effect of adsorbent dosage on the adsorption of fluoride ion was studied using different bottom ash dosage in the range of 10-100 mg/100ml (Fig.2) at 293 K, 303 K and 313 K. Results showed that the adsorption efficiency was highly dependent on the increase in bottom ash dosage of the solution. Fluoride uptake was very low at low bottom ash dosage and increased with dosage. This is due to a

low dose, all types of sites are entirely exposed and the adsorption on the surface is saturated faster, showing a higher q_e value. But at a higher adsorbent dose, the availability of higher energy sites decreases with a larger fraction of lower energy sites occupied, resulting in a lower q_e value (Gupta et al., 2010). The equilibrium bottom ash dosage decreases with the temperature. The dosage is varying according to the temperature as 100mg for 293 K, 80 mg for 303 K and 60 mg for 313 K for 100ml fluoride solution.

Effect of Particle Size

Experiments were conducted to evaluate the influence of adsorbent particle size for a constant weight on the removal of fluoride ions. The uptake of fluoride ions at different adsorbent particle sizes increased with a decrease in an adsorbent particle diameter (Fig. 3). This is mainly true since the surface area and the number of active pores of the adsorbent increase with the decrease in particle size. Solute adsorption onto the adsorbent is also dependent on pore size distribution and will depend on the number of micro-, meso-, and macropores in the structure (Streat et al. 1995). The access to all pores is facilitated in small size particles (Gupta et al. 2010).

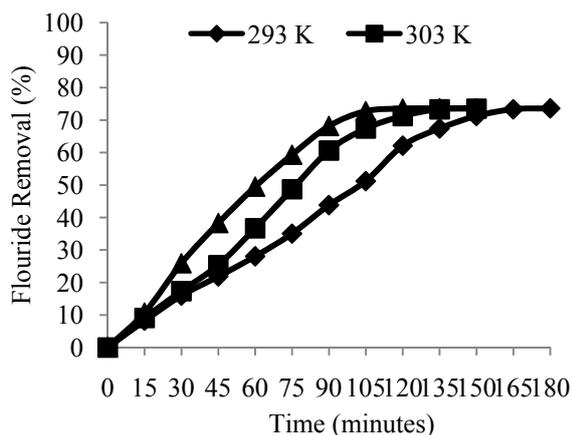


Fig. 1 Effect of contact time and temperature on adsorption of fluoride by bottom ash

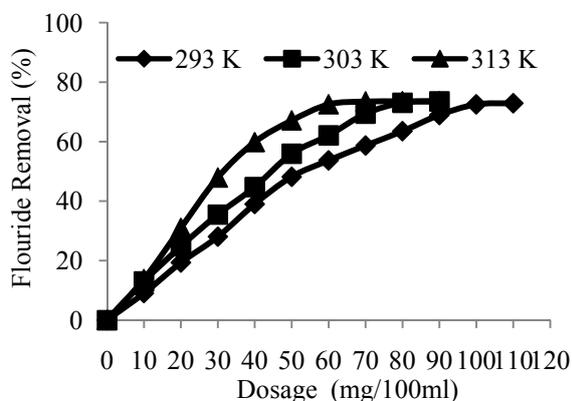


Fig. 2. Effect of bottom ash dosage on fluoride removal

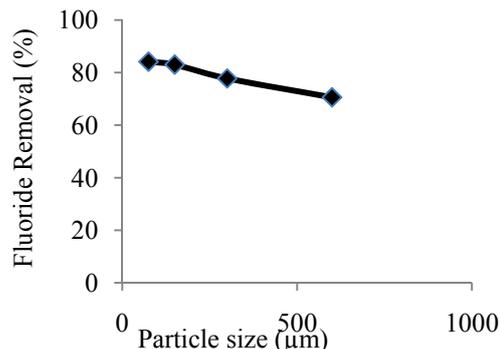


Fig. 3. Effect of particle size on adsorption of fluoride by bottom ash

Effect of pH

The pH of the aqueous solution is a controlling factor in the adsorption process. Thus, the role of hydrogen ion concentration was examined at pH values of 4 - 10. This was adjusted by adding 0.5 N HNO₃ or 0.1 M NaOH with 50 ml of standard solution of 10 mg/l of fluoride for a contact time of 90 min with a dose of 80 mg/100ml of adsorbent. The influence of pH on the sorption rate is shown in Fig. 4. Equilibrium fluoride sorption increases with pH up to neutral and decreases with increase in pH. Maximum adsorption by the bottom ash was observed at pH 6.0.

Effect of Rotation

For the effect of rotation on fluoride removal optimum dose of 80 mg/100ml used of the influent solution and the solution were then taken in conical flasks and kept in the orbital shaking incubator where the rotations manually changed. Three agitation speeds 100, 150 and 200 rpm were tested in order to investigate the kinetic process of fluoride adsorption and the results are illustrated in Fig. 5. It was observed that at 100rpm the equilibrium time was found to be 165 min as same as that of 200 rpm. But at 150 rpm the equilibrium time was found to be less than other two speeds (135 min).

Isotherm Study

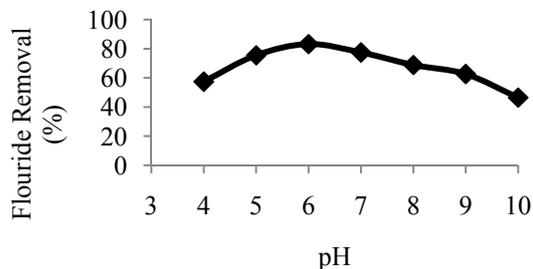


Fig. 4. Effect of pH on Fluoride removal by Bottom ash

The adsorption data were analyzed by a regression analysis to fit the Freundlich (Freundlich 1906) and Langmuir (Langmuir 1915) isotherm models. The Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number

of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface (Fytianos et al. 2000). While Freundlich isotherm model assumes heterogeneous surface energies, in which the energy term in the Langmuir equation varies as a function of the surface coverage (Weber and Chakkravorti 1974). The Freundlich and Langmuir adsorption isotherm plotted for fluoride ion are shown in Figs. 6 and 7. The Langmuir and Freundlich adsorption constants evaluated from the

isotherms together with the correlation coefficients are presented in Table 1. Regression values (R^2) indicates that the adsorption data for fluoride onto bottom ash fitted well with both isotherm models. The adsorption capacity of bottom ash increases with temperature. The value of Langmuir constant 'b' decreases as the temperature increases. Freundlich constant (n) values are in the range of 1–10 for all the temperature, which indicates favourable adsorption.

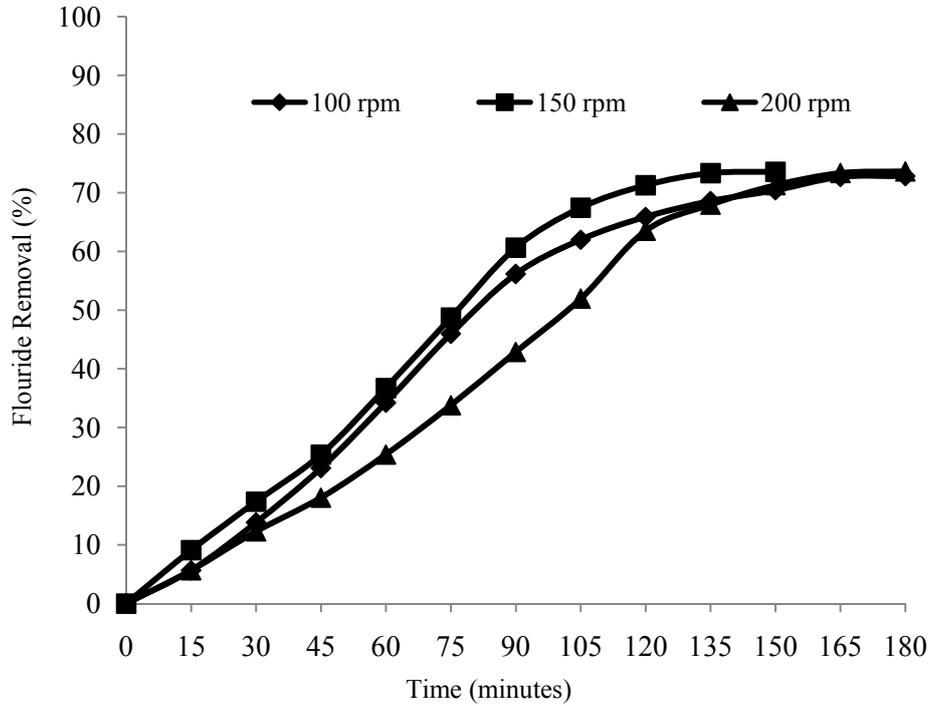


Fig. 5. Effect of rotation on fluoride removal

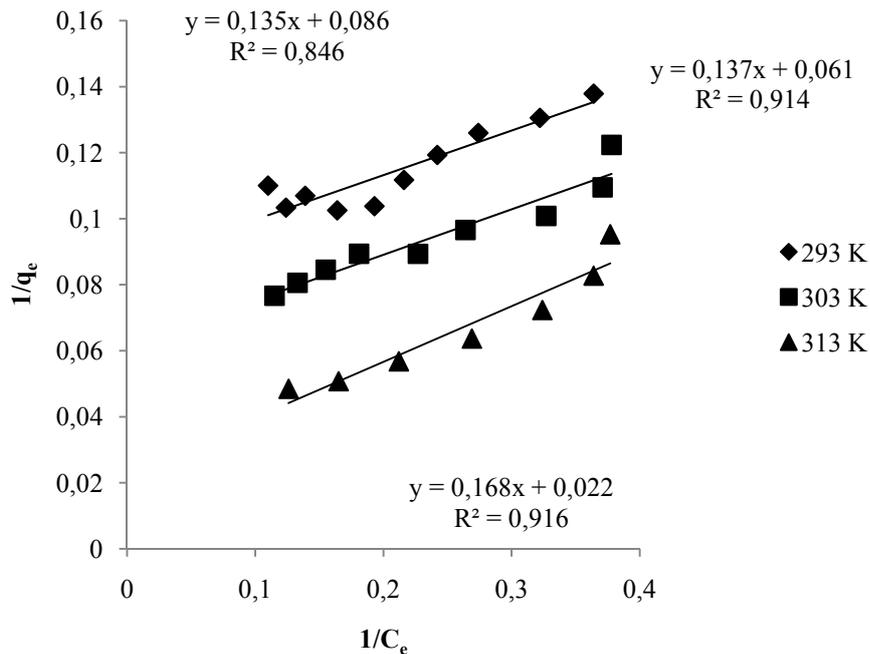


Fig. 6. Langmuir isotherm plot for fluoride removal

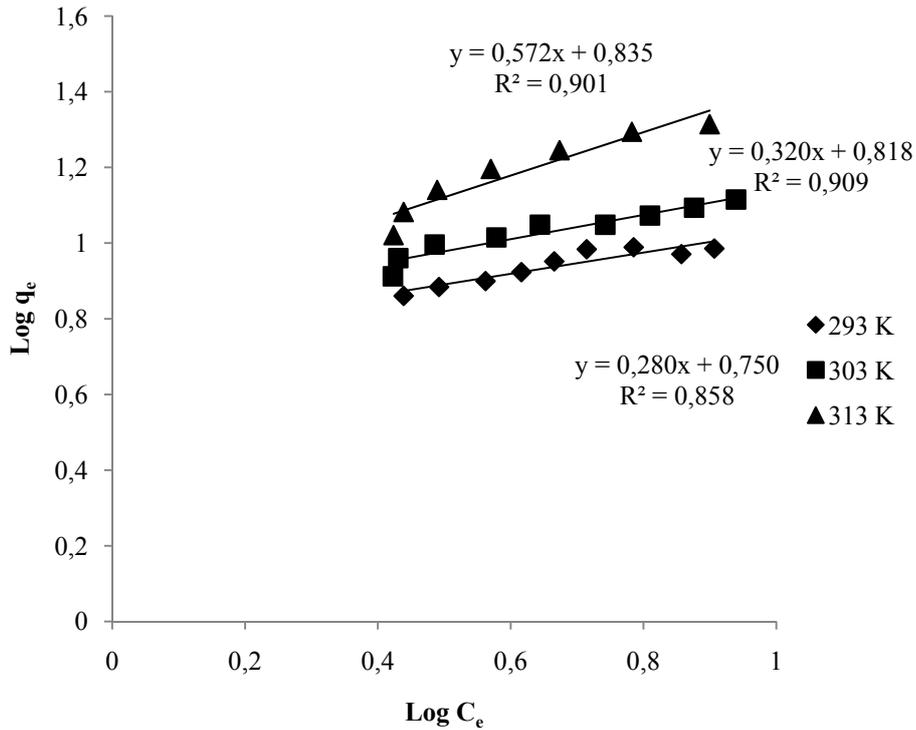


Fig.7. Freundlich Isotherm Plot for Fluoride Removal

Table 1. Isotherm Constants for adsorption of Fluoride

Parameters	Temperature (K)		
	293	303	313
Langmuir isotherm			
q_{max} (mg/g)	11.60	16.26	43.69
b	0.638	0.446	0.137
R^2	0.846	0.915	0.916
Freundlich isotherm			
K_f (mg/g)	5.631	6.579	6.839
n	3.565	3.120	1.746
R^2	0.858	0.909	0.901

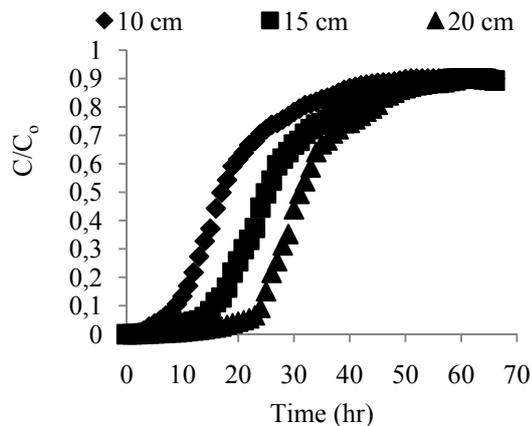


Fig. 8. Breakthrough Curves at Different Bed Depths at 10 ml/min Flow Rate

Column Study

The column experiments were conducted with 10 mg/l fluoride solutions at bed depths of 10 cm, 15 cm, and 20 cm, respectively, at a constant flow

rate of 10 ml/min. The breakthrough curves of S shape were obtained as shown in Fig. 8. It was observed that the time to achieve breakthrough increased with an increase in bed depth.

Evaluation of Column Design Parameters

Data collected during laboratory tests serve as the basis for the design of full-scale adsorption columns. By interpolation from the above values, the service time corresponding to the three bed depths can be found. These are listed below in Table 2.

Table 2. Service Time at Different Bed Depths

S. No.	Bed Depth (cm)	Service Time (hr)
1	10	6.06
2	15	13.39
3	20	21.24

A plot between service time and bed depth is now drawn using the BDST model (Eq. 1) (Bohart and Adams 1920). where C_0 - initial concentration of solute (mg/l), C_B - desired concentration of solute at breakthrough (mg/l), k_a - adsorption rate constant (l/mg/ h), N_0 - adsorption capacity (mg/l), H - bed depth of column (cm), u - linear flow velocity of feed to bed (cm/hr), t - service time of column under above conditions (hr). The critical bed depth (H_0) is the theoretical depth of adsorbent sufficient to ensure that the outlet solute concentration does not exceed the breakthrough concentration (C_B) value at time $t=0$. H_0 can be calculated as Eq. (2) (Sarin et al. 2006). The line of a best fit for the plot is obtained by the method of least squares. The obtained graph is shown in Fig. 9.

$$t = \frac{HN_0}{u C_0} - \frac{\ln(C_0/C_B - 1)}{k_a C_0} \quad (1)$$

$$H_0 = \frac{u}{N_0 k_a} \ln\left(\frac{C_0}{C_B} - 1\right) \quad (2)$$

The equation of fitted line is found to be:

$$t = 1.518H - 9.206 \quad (3)$$

The value of R^2 for the best fit line is found to be 0.999

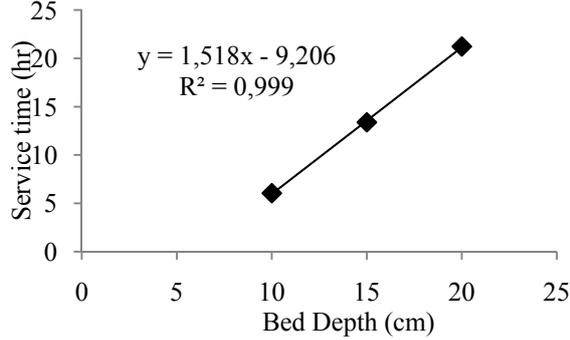


Fig. 9. BDST Plot for flow rate of 10 ml/min

From the above graph, the column design parameters are calculated, and are shown in Table 3.

Table 3. BDST Parameters for 10 ml/min Flow Rate

S. No.	Parameter	Value
1	K	57.78 cc/mg-hr
2	N_0	0.578 mg/cc
3	H_0	3.248 cm

Thomas Model

Successful design of a column adsorption process requires prediction of the concentration versus time profile or breakthrough curve for the effluent. The maximum sorption capacity is also in design. Traditionally, the Thomas equation is employed to fulfill the purpose (Qing 2010). The expression of the Thomas model for an adsorption column is given below (Sivakumar and Palanisamy 2009):

$$\frac{C}{C_0} = \frac{1}{\left[1 + \exp\left(\frac{K_T(qm - C_0V)}{r}\right)\right]} \quad (4)$$

Substituting $t = V/r$ in Eq. (4), it becomes

$$\frac{C}{C_0} = \frac{1}{\left[1 + \exp\left(K_T\left\{\frac{qm}{r} - C_0t\right\}\right)\right]} \quad (5)$$

It can be rewritten as

$$K_T\left(\frac{qm}{r} - C_0t\right) = \ln\left(\frac{C_0}{C} - 1\right) \quad (6)$$

This is in the form of

$$Y = b - at \quad (7)$$

Having slope $a = K_T C_0$, intercept $b = \frac{K_T qm}{r}$

Where C is the effluent concentration (mg/l), C_0 is the influent concentration (mg/l), K_T is the Thomas rate constant (l/min.mg), q is the maximum adsorption capacity (mg/g), m is the mass of adsorbent (g), V is the effluent volume (ml) and r is the flow rate (ml/min). Constants K_T and q can be determined from a plot of $\ln(C/C_0 - 1)$ against t (Sivakumar and Palanisamy 2009). Fig. 10 shows the Thomas model plot between $\ln(C/C_0 - 1)$ against t and the constants are shown in Table 4.

Table 4. Thomas model constants of Fluoride Removal by Bottom ash

Bed Depth (cm)	K_T (l/min.mg)	q (mg/g)
10	0.0619	0.3714
15	0.0614	0.3684
20	0.0044	0.0264

Yoon Nelson Model

The Yoon–Nelson is based on the assumption that the rate of decrease in the probability of adsorption for each adsorbate molecule is proportional to the probability of adsorbate adsorption and the probability of adsorbate breakthrough on the adsorbent (Han et al. 2009). The Yoon–Nelson equation for a single component system is expressed as (Aksu and Gönen 2004)

$$\frac{C}{C_0} = \frac{1}{1 + \exp(K_{YN}\{\tau - t\})} \quad (8)$$

This can be rewritten as

$$\ln\frac{C}{C_0 - C} = K_{YN}(t - \tau) \quad (9)$$

This is in the form of

$$Y = at - b \quad (10)$$

Where slope $a = K_{YN}$ and intercept $b = K_{YN} \tau$

Where τ is the time required for 50% adsorbate breakthrough (min), K_{YN} is the Yoon Nelson rate constant (1/min). Both constants can be found out

from a plot of $\ln [C/(C_0 - C)]$ against t. Fig.11 shows the Yoon Nelson plot between $\ln [C/(C_0 - C)]$ against t, the constants are in Table 5.

Table 5. Yoon Nelson constants of Fluoride Removal by Bottom ash

Bed Depth (cm)	K_{YN} (l/min)	τ (minutes)
10	0.003	943.16
15	0.004	1468.50
20	0.006	2139.67

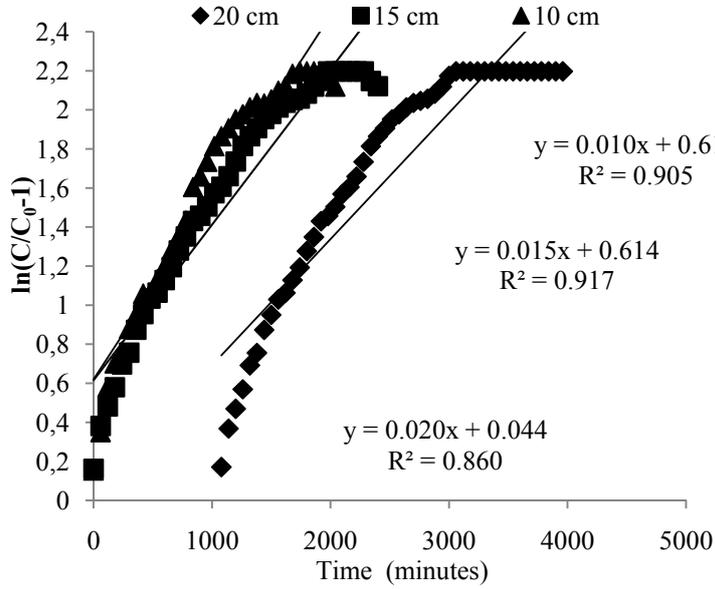


Fig. 10. Thomas Model Plot for Fluoride Removal by Bottom ash

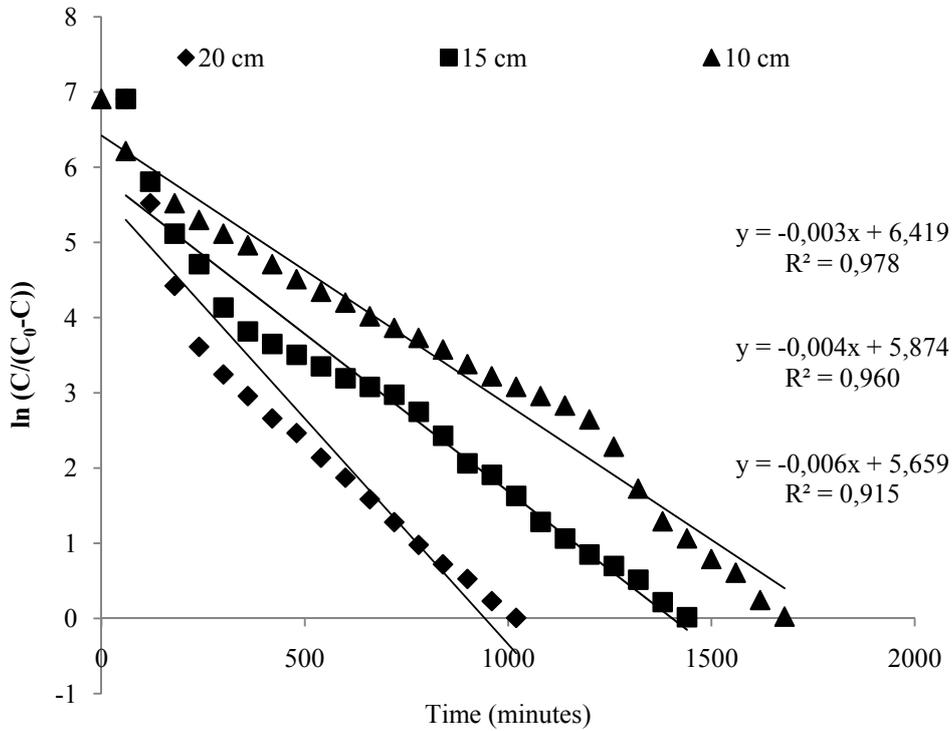


Fig. 11. Yoon Nelson Model Plot for Fluoride Removal by Bottom ash

4. Conclusions

The present work deals with the use of batch and continuous fixed bed column studies on adsorption of fluoride on bottom ash. The optimum contact time for fluoride was found to be 105 minutes with the maximum efficiency of 73.5 % at 70mg/100ml bottom ash dosage. The optimum pH was found to be pH 6 with the maximum efficiency of 83.2 %. Also the fluoride removal increased with a decrease in the particle size. The maximum monolayer adsorption capacity of bottom ash was found to be 16.26 mg/g at 303 K. From the column study, it was found that the increase in fluoride ion uptake with an increase in the bed height was due to an increase in the contact time. A high degree of linearity of the BDST plot indicates the validity of the BDST Model when applied to continuous column studies. From the Thomas model, it is observed that maximum adsorption capacity is found 0.3714 mg/g and the rate constant is 0.0619 l/min.mg. From the Yoon Nelson model it is observed that 50% of adsorbate time 2140 minutes run at the rate constant of 0.003 l/min.

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Dr. S.T. Ramesh, Associate Professor at Department of Civil Engineering, National Institute of Technology, India.
Main research area: Adsorption, Water and wastewater treatment, Solid waste management
Address: Department of Civil Engineering,
National Institute of Technology,
Tiruchirappalli, Tamilnadu,
India 620015
Tel.: +91 431 250 3166
E-mail: sramesh@nitt.edu

P.V. Nidheesh Ph.D. Scholar at Department of Civil Engineering, National Institute of Technology, India.
Main research area: Physico Chemical Treatment of water and wastewater treatment, Solid waste management
Address: Department of Civil Engineering,
National Institute of Technology,
Tiruchirappalli, Tamilnadu,
India 620015
Tel.: 07373952468
E-mail: nidheeshpv129@gmail.com

Dr. R. Gandhimathi, Assistant Professor at Department of Civil Engineering, National Institute of Technology, India.
Main research area: Adsorption, Water and wastewater treatment, water quality modelling
Address: Department of Civil Engineering,
National Institute of Technology,
Tiruchirappalli, Tamilnadu,
India 620015
Tel.: +91 431 2503171
E-mail: rgmathii@nitt.edu

Manoj Taywade M.Tech Student at Department of Civil Engineering, National Institute of Technology, India.
Main research area: water and wastewater treatment, Adsorption Process
Address: Department of Civil Engineering,
National Institute of Technology,
Tiruchirappalli, Tamilnadu,
India 620015

Pelenų naudojimas fluoridui šalinti iš vandens

S. T. Ramesh, R. Gandhimathi, P. V. Nidheesh, M. Taywade

Statybų inžinerijos katedra, Valstybinis technologijų institutas, Indija

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Milijonams žmonių tiekiamas vanduo, kuriame yra per didelis fluorido kiekis. Vietovėse, ypač mažose bendruomenėse, kuriose didelė fluorido koncentracija, jo šalinimas iš geriamo vandens vis dar yra problema. Straipsnyje aprašytas tyrimas, kurį atliekant vertinta pelenų adsorbicija fluorido ir vandens šalinimo procese.