



A study on removal of fluoride ions using Aloe Barbadensis as a low- cost natural adsorbent

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ABSTRACT

The study assessed the suitability of low-cost natural adsorbent to effectively remediate fluoride contaminated water. The removal of fluoride from aqueous solution by using Aloe Barbadensis was studied in batch technique. Influence of pH, adsorbent dose, contact time, co ions, speed and initial concentration on the adsorption were investigated. The maximum removal of fluoride ion was obtained at pH 7. The removal of fluoride was expressed with Langmuir and Freundlich isotherm. It was found that the sufficient time for adsorption equilibrium of fluoride ion is 1 hour. The removal of fluoride ions was maximum for the adsorbent dosage of AB is 10mg 50mL⁻¹. The fluoride adsorption was maximum at 60min. The adsorption of F ion was maximum in the shaking speed of 120 rpm. The presence of interfering ions such as nitrate showed positive effect while carbonate, sulphate, chloride showed little negative effect and phosphate showed high negative effect for the adsorbent. The optimum initial F concentration for AB adsorbent was 5mg 50mL⁻¹.

Keywords: Fluoride, AB-Aloe Barbadensis, low-cost natural adsorbent.

INTRODUCTION

Water is an essential natural resource for sustaining life and environment that we have always thought to be available in abundance and free gift of nature. However, chemical composition of surface or subsurface is one of the prime factors on which the suitability of water for domestic, industrial or agricultural purpose depends. Freshwater occurs as surface water and groundwater. Though groundwater contributes only 0.6% of the total water resources on earth, it is the major and the preferred source of drinking water in rural as well as urban areas, particularly in the developing countries like India because treatment of the same, including disinfection is often not required. It caters to 80% of the total drinking water requirement and 50% of the agricultural requirement in rural India. But in the era of economical growth, groundwater is getting polluted due to urbanization and industrialization. Over the past few decades, the ever-growing population, urbanization, industrialization and unskilled utilization of water resources have led to degradation of water quality and reduction in per capita availability in various developing countries. Due to various ecological factors either natural or anthropogenic, the groundwater is getting polluted because of

deep percolation from intensively cultivated fields, disposal of hazardous wastes, liquid and solid wastes from industries, sewage disposal, surface impoundments etc. [1–4]. During its complex flow history, groundwater passes through various geological formations leading to consequent contamination in shallow aquifers. Presence of various hazardous contaminants like fluoride, arsenic, nitrate, sulphate, pesticides, other heavy metals etc. in underground water has been reported from different parts of India [5–12]. In many cases, the water sources have been rendered unsafe not only for human consumption but also for other activities such as irrigation and industrial needs. Therefore, now there is a need to focus greater attention on the future impact of water resources planning and development taking into consideration all the related issues. In India, fluoride is the major inorganic pollutant of natural origin found in groundwater. So far various researchers have used plant materials as adsorbents for the removal of fluoride ions [13 – 25].

The aim of the present study was to examine the feasibility of using low cost natural adsorbents such as *Aloe Barbadensis*, in Fluoride removal using adsorption technique. Adsorption characterisation was done by using SEM, XRD and FT-IR techniques. The effect of adsorbent dosage, contact time, pH, co-ions, initial fluoride concentration and shaking speed were determined.

MATERIALS AND METHODS

Fluoride Adsorption Experiments: A stock solution (100 mg L^{-1}) was prepared by dissolving 221mg NaF (analytical grade) in 1 L of distilled water. All the solutions for fluoride removal experiments and analysis were prepared by an appropriate dilution from the stock solution. Adsorption studies were carried out for each desired initial fluoride concentration solution in a conical flask immersed in a temperature-controlled water bath and shaken with a mechanical shaker (Tai Tec, Thermo Minder Mini-80, Japan) for the required time at a rate of 120 cycles/min. Fluoride analysis was carried out with photocolourimetry using the SPADNS indicator. The adsorption isotherm at $\text{pH } 6.90 \pm 0.10$ was studied by varying the initial fluoride concentration from 5 to 50 mg L^{-1} . The effect of pH was investigated by adjusting the pH from 2 to 12 using 0.1 M NaOH and HCl solutions under an initial fluoride concentration of 10 mg L^{-1} . A kinetic study at $\text{pH } 6.90 \pm 0.10$ was carried out at different time intervals with an initial fluoride concentration of 10 mg L^{-1} . Finally, the effects of a number of competing ions (Cl^- , NO_3^- , CO_3^{2-} , SO_4^{2-} , and PO_4^{3-}) were observed using, 20-200 mg L^{-1} solutions at $\text{pH } 6.90 \pm 0.10$ over 1 h.

Physical characterizations: The surface morphology and size distribution of the ayurvedic adsorbent powders were observed by means of a SEM, XRD and FTIR spectral analysis. The ayurvedic adsorbent powders before and after treated batch adsorption experiments was characterized using XRD and SEM. A representative experiment was carried out under the given conditions: 30°C , initial F- concentration =50 mg/L, shaking speed =150 rpm and contact time=1 h respectively, for the preparation of XRD and SEM samples. The X-ray diffraction patterns were recorded using X-ray diffractometer, Model (Phillips, 'X' Pert). The sample was scanned for 2θ range from 0° to 60° . The SEM analysis was carried out using Jeol, XA-840 A. The FTIR spectra were recorded on a Nicolet 560 FTIR spectrophotometer.

Chemicals Used : Stock solution of fluoride was prepared by dissolving 221mg of sodium fluoride in 1 L distilled water. The measuring cylinder, volumetric flask and conical flask used. The sodium fluoride used was an analytical grade (Merck, Germany). The required concentration of fluoride solution was prepared by serial dilution of 100 mg/L Fluoride solution. Indicator was SPANDS reagent.

Theory of Isotherm Models : The abilities of four widely used isotherms, the theoretical Langmuir, empirical Freundlich, Temkin and Redlich-Peterson isotherms, to model the adsorption equilibrium data were examined. To express the mechanism of fluoride adsorption onto the surface of adsorbent, the kinetic models pseudo first order, pseudo second order, intra particle diffusion and Elovich models are used to analyze the present adsorption data to determine the related kinetic parameters. Langmuir adsorption

isotherm is perhaps the best known of all isotherms which is often applied in solid/liquid system to describe the saturated monolayer adsorption. It can be represented as:

$$q_e = \frac{q_m K_a C_e}{1 + K_a C_e} \quad (1)$$

Where C_e is the equilibrium concentration (mg L^{-1}); q_e is the amount of ion adsorbed (mg g^{-1}); q_m is q_e for a complete monolayer (mg/g); K_a is adsorption equilibrium constant (mg L^{-1}). To evaluate the adsorption capacity for particular range of adsorbate concentration, the before mentioned equation Eq. (1) can be used as a linear form as follows :

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_a q_m} \quad (2)$$

The constants q_m and K_a can be determined from a liberalised form of Eq. (2) by the slope of the linear plot of C_e/q_e versus C_e .

Freundlich adsorption isotherm based on adsorption on heterogeneous surface is the earliest known relationship describing the adsorption equilibrium and is given by:

$$q_e = K_F C_e^{1/n} \quad (3)$$

where q_e is the amount of ion adsorbed (mg/g); C_e is the equilibrium concentration (mg L^{-1}); K_F and $1/n$ are empirical constants, indicating the adsorption capacity and adsorption intensity, respectively. The Eq. (3) may be converted to a linear form by taking logarithms.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

The plot of $\log q_e$ versus $\log C_e$ of Eq. (4) should result in a straight line. From the slope and intercept of the plot, the values for n and K_F can be obtained.

Temkin Isotherm, the simple form of adsorption isotherm model, has been developed considering the chemisorptions of an adsorbate onto the adsorbent, is represented as

$$q_e = a + b \log C_e \quad (5)$$

where q_e and C_e have the same meaning as noted previously and the other parameters are called the Temkin constants. The plot of q_e versus $\log C_e$ will generate a straight line. The Temkin constants a and b can be calculated from the slope and intercept of the linear plot.

Redlich-Peterson isotherm contains three parameters and incorporates the features of the Langmuir and the Freundlich isotherms. It can be described as follows:

$$q_e = \frac{AC_e}{1 + BC_e^g} \quad (6)$$

Eq. (6) can be converted to a linear form by taking natural logarithms:

$$\ln \left(A \frac{C_e}{q_e} - 1 \right) = g \ln (C_e) + \ln (B) \quad (7)$$

Three isotherm constants, A , B , and g ($0 < g < 1$), can be evaluated from the linear plot represented by Eq. (7) using a trial and error optimization method.

RESULTS AND DISCUSSION

Fluoride removal by adsorbent *Aloe Barbadensis*

Effect of Adsorbent Dose: The influence of adsorbent dose on fluoride removal at a fixed initial fluoride concentration of 100mg/litre and $\text{pH } 7$ is shown in figure 1. It was noticed that percentage removal of fluoride decreased from 96% to 60% with an increase in adsorbent dose from $10\text{-}200\text{mg } 50\text{mL}^{-1}$ respectively. However, It was noticed that after a dosage of $150 \text{ mg } 50\text{mL}^{-1}$ there was no significant change in the percentage of removal. It may be due to overlapping of active sites at higher dosage, thus decreasing the net surface area of the adsorbent [26]. So an adsorbent dose of $10\text{mg } 50\text{mL}^{-1}$ was used for further study.

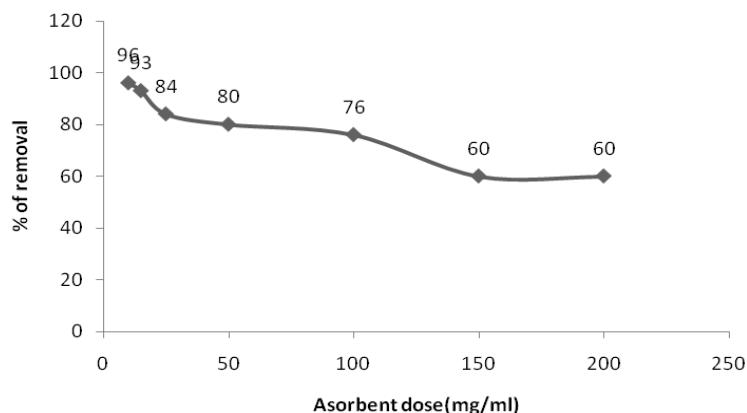


Fig.1 Effect of adsorbent dosage

Effect of Contact Time: The results of equilibrium studies are shown in figure 2. Contact time experiments showed that the removal of fluoride ions increased with time up to 60min and after which the increase in agitation time did not alter the fluoride ion uptake due to attainment of equilibrium. Fluoride ion uptake by the adsorbents indicated that the process was rapid with typically 10mg 50mL⁻¹ of *Aloe barbadensis* with 60min of contact time. The effect of contact time is shown in figure 2.

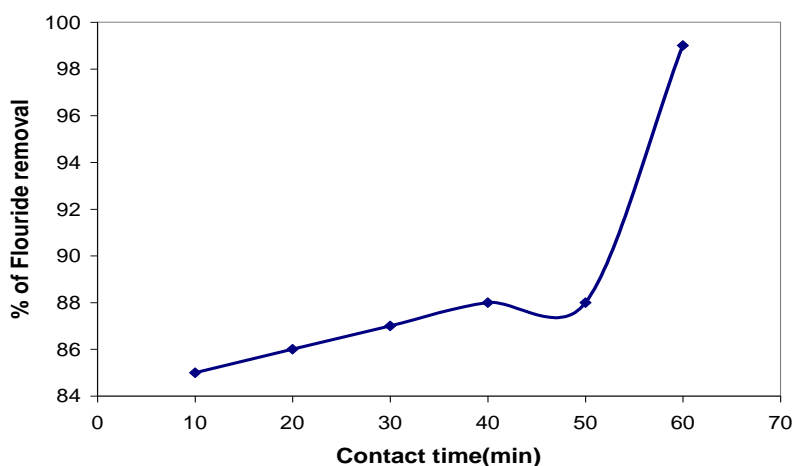


Fig.2 Effect of contact time

Effect of pH: The effect of pH on fluoride removal by AB was assessed using pH sweep experiments, where powder of 10mg 50mL⁻¹ and 0.01 ppm 50mL⁻¹ of fluoride concentration was reacted for 1 h with different amounts of HCl/ NaOH solution. Fig.3 shows that fluoride removal reactions in AB powder are dependent on pH. The pH of the medium is one of the important parameters that significantly affect the extent of F⁻ adsorption. F⁻ removal by the adsorbent AB was studied over the pH range of 3 to 11.5 with an adsorbent dose = 10mg/50ml, initial F conc. = 100mgL⁻¹, shaking speed = 120 rpm and contact time = 1h as shown in fig.3. It was evident from the results that the F removal was low at low pH. As the pH increased up to 7, the fluoride removal increased and further increase of pH, the fluoride removal dropped down. It might be because of the competition between OH⁻ and F⁻ ions in high pH range [3].

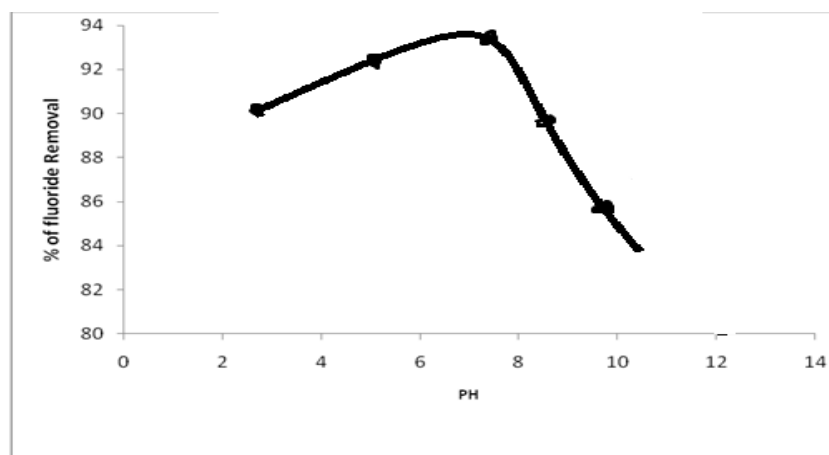


Fig.3 Effect of pH

Effect of Co-Ions on F⁻ Adsorption:

Table – 1 Effect of Co-ions

No	Types of salts	% of F ⁻ removal
1	Blank	96
2	NaNO ₃	96
3	NaCO ₃	93
4	NaCl	88
5	Na ₂ SO ₄	93
6	PO ₄ ³⁻	13

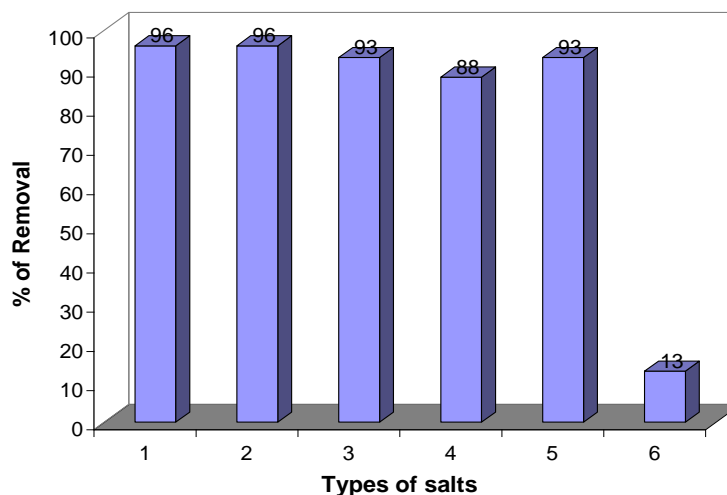


Fig.4 Effect of co-ions

To study the influence of interfering ions, fluoride removal experiments were conducted in the presence of salt solutions of nitrate, carbonate, chloride, sulphate and phosphate separately. Dose = 10mg 50mL⁻¹, shaking speed = 120 rpm and contact time is 1h. The results are shown in table1 and fig.4. It was noticed that only nitrate showed positive effect while carbonate, sulphate, chloride and phosphate showed negative effect on fluoride removal by AB powder. This might be due to a change in pH and also because of competition between the competing ions and fluoride ions for the active site of AB Powder [26]

Effect of Speed

Effect of shaking speed was studied over range of 100-300 rpm is shown in fig.5. From fig.5, it has been determined that the shaking speed of 120 rpm was sufficient to remove maximum F⁻ ions from the solution. This might be due to the increase of kinetic energy of F⁻ ions at 120 rpm speed. Collision frequency between adsorbent and adsorbate was found to be maximum at 120 rpm.

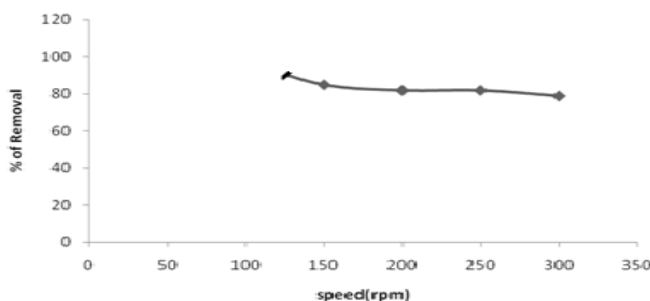


Fig.5 Effect of speed

Effect of Initial Fluoride Concentration

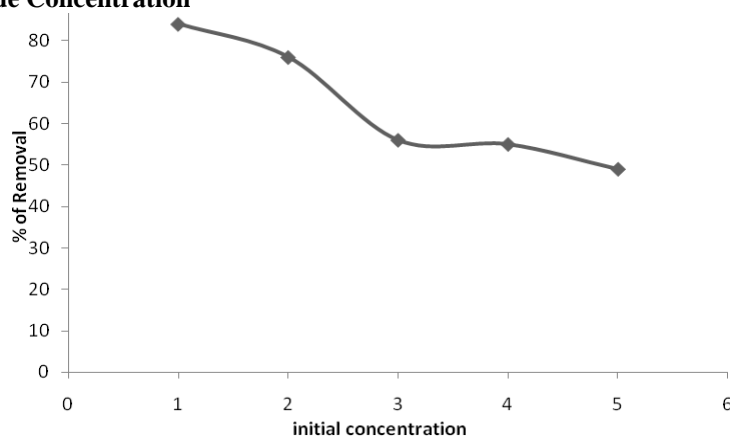


Fig.6 Effect of Initial Fluoride Concentration

The influence of initial fluoride concentration on fluoride removal from aqueous solution was studied by keeping all other parameters constant i.e., dose = $10\text{mg } 50\text{mL}^{-1}$, contact time = 1h and shaking speed = 120 rpm as shown in fig 6. It was noticed that with increase in initial fluoride concentration, the percentage removal of fluoride decreased. This suggests that there existed a reduction in immediate solute adsorption due to the lack of available active sites on the adsorption surface, compared with the relatively large number of active site required for the high initial concentration of fluoride [26].

Adsorbent Characteristics: The XRD & SEM patterns of AB before (untreated) and after treatment (treated) with F^- ions are shown in Fig.7 **a** and **b** and XRD pattern before and after treatment are given in the fig. 8 **a** and **b**. It is evident from the XRD studies that the crystal structure of the AB showed significant changes after the adsorption of F^- ions. It is evident from the pattern that the adsorbent possessed predominantly amorphous character and to a very extent crystalline character also. The XRD pattern of AB loaded with fluoride ions showed significant changes when compared to that of AB before adsorption. This suggested that the uptake of fluoride ions by AB is by chemisorptions which consequently alters the structure of the adsorbent.

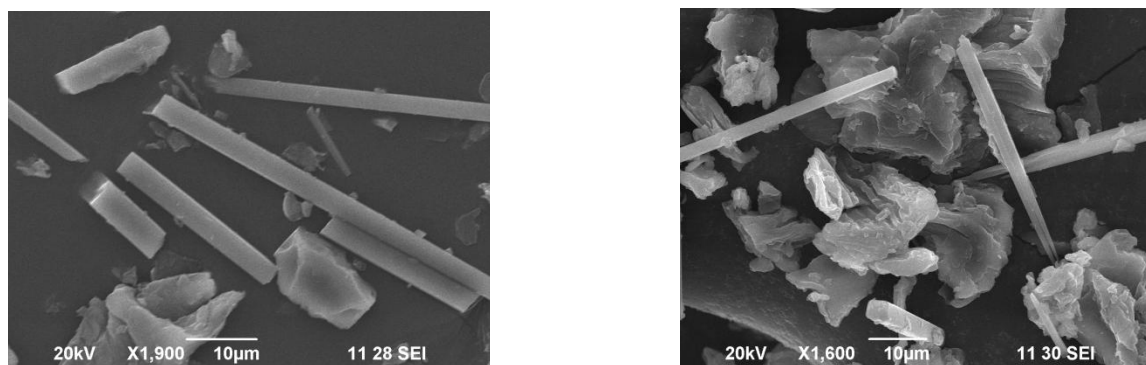


Fig.7 a. SEM images of untreated AB, b. treated AB

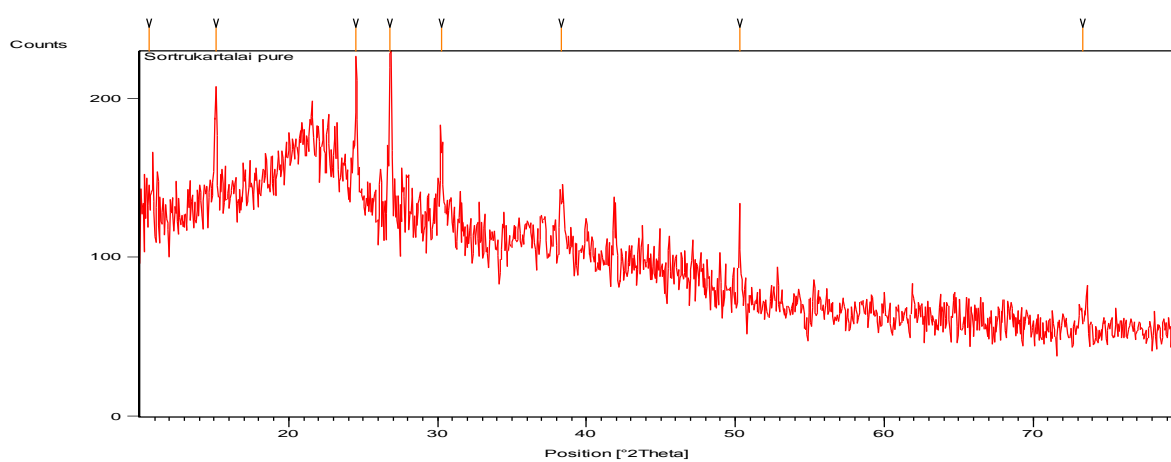


Fig. 8 a. XRD pattern before treatment of AB

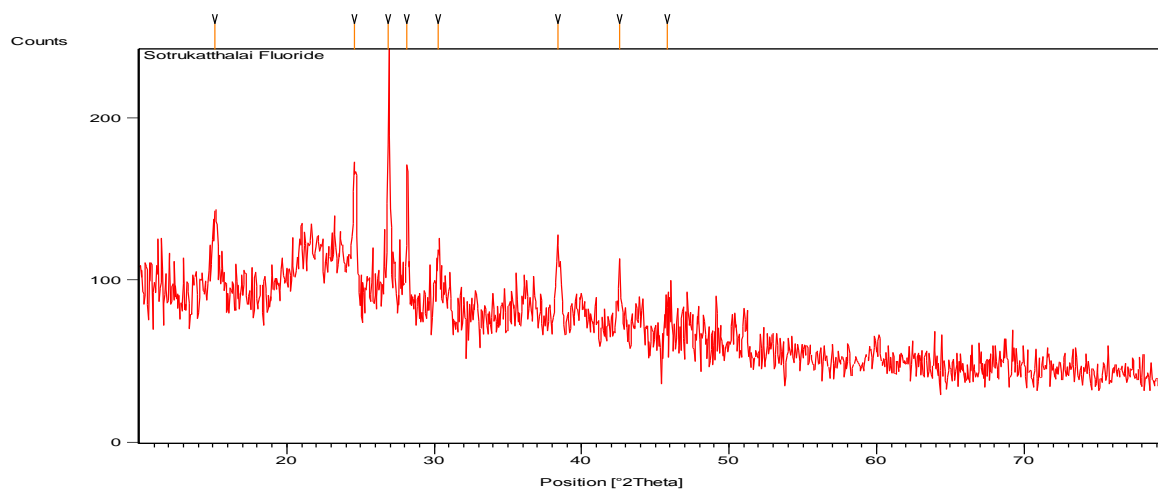


Fig. 8 b. XRD pattern after treatment of AB

Adsorption Isotherm

The experimental data was fitted to Freundlich, Langmuir, Dubinin and Radhushkevich (R-D) and Temkin isotherm models.

The linearized Freundlich adsorption isotherm is of the form

$$\log q_e = \log k_f + 1/n (\log C_e) \quad (8)$$

where q_e is the adsorbed fluoride at equilibrium per unit mass of adsorbents (mg/g), k_f the minimum sorption capacity (mg g⁻¹) and $1/n$ is the adsorption intensity, C_e is the equilibrium concentration of fluoride (mg L⁻¹).

The Langmuir adsorption isotherm equation is represented as:

$$q_e = q_m \times (BC_e / (1+BC_e)) \quad (9)$$

and the linearized form can be represented as:

$$1/q_e = 1/q_m + 1/Bq_m (1/C_e) \quad (10)$$

where q_e is the amount of solute adsorbed per unit weight of material (mg g⁻¹), q_m the maximum adsorption capacity (mg/g), B the Langmuir constant and C_e is the equilibrium solute concentration. The essential characteristic of the Langmuir isotherm can be expressed by the dimensionless constant called equilibrium parameter, R_L , defined by:

$$R_L = 1/(1+BC_0) \quad (11)$$

Where B is the Langmuir constant and C_0 is the initial adsorbate concentration (mg/L), R_L values indicate the type of isotherm to be irreversible ($R_L = 0$), favourable ($0 < R_L < 1$), linear ($R_L = 1$) or unfavourable ($R_L > 1$). The R_L value for the adsorption is shown in Table 2.

Another equation used in the analysis of isotherms was the Dubinin and Radushkevich model:

$$Q_e = q_s \exp(-B\varepsilon^2) \quad (12)$$

where q_s is the D-R constant. The sorption data was modelled by D-R isotherm to determine the adsorption type (physical or chemical). The linear form of this model is expressed by:

$$\ln q_e = \ln q_s - \beta \varepsilon^2 \quad (13)$$

where q_e is the amount of fluoride adsorbed onto the surface of the adsorbent per unit dosage of the adsorbent (mol g⁻¹), q_m the monolayer capacity (mol/g), C_e the equilibrium fluoride concentration (mol L⁻¹) and β is the activity coefficient related to mean sorption energy (mol² kJ²⁻¹). E is the Polanyi potential described as:

$$\varepsilon = RT \ln (1+(1/C_e)) \quad (14)$$

The mean sorption energy, E (kJ/mol) can be calculated as:

$$E = 1/(-2 \beta)^{1/2} \quad (15)$$

The Temkin isotherm is given as:

$$q_e = (RT/b) \ln (K_T C_e) \quad (16)$$

which can be linearized as:

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (17)$$

where

$$B_1 = RT/b \quad (18)$$

Temkin isotherm contains a factor that explicitly takes into the account adsorbing species and adsorbent interactions. The heat of adsorption of all the molecules in the layer would decrease linearly with coverage due to adsorbate/adsorbent interactions [4]. This isotherm assumed that (i) the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent and adsorbate interactions and that (ii) the adsorption is characterized by a uniform distribution of binding energies up to some maximum binding energy. A plot of q_e versus $\ln C_e$ enables the determination of the isotherm constants B_1 and K_T from the slope and intercept, respectively. K_T is the equilibrium binding constant (mol L⁻¹) corresponding to the maximum binding energy and constant B_1 is related to the heat of adsorption.

Fig.9 shows a plot of $\log q_e$ versus $\log C_e$. The constants $1/n$ and $\log k_f$ can be obtained from slope and intercept respectively. It is found that the related correlation coefficient R^2 value for the Freundlich model is 0.9824. Freundlich isotherm shows the minimum adsorption capacity of 0.94 mg/g of the adsorbent. The

experimental data fit well to the Freundlich isotherm model as well. The condition for the validity of a Freundlich type adsorption model is adsorption on heterogeneous surfaces.

In fig.10, plots of $1/q_e$ versus $1/C_e$ yield straight lines demonstrating that Langmuir isotherm fits the sorption data very well. The value of q_m is 40 mg/g and the Langmuir constant B is 0.0235 mg L^{-1} . The results are summarized in Table.3. The value of R_L , 0.2985 also indicates favourable sorption of fluoride on to the adsorbent.

Fig.12 shows the Temkin isotherm plot for the adsorption of fluoride and the corresponding constants are presented in Table.2. A linear relationship between q_e and $\ln C_e$ indicates the applicability of this model to understand the adsorption mechanism. The corresponding constants are given in Table.2. fig.11 shows the R-D plot for the adsorption of fluoride and the corresponding constant are presented in table.3. The correlation coefficient for the Langmuir isotherm is highest in comparison to the values obtained for the Freundlich, Temkin and Dubinin and Radushkevich isotherms. Therefore, the Langmuir isotherm is the best-fit isotherm for the adsorption of fluoride onto this adsorbent, AB under the experimental conditions used in this study. The monolayer sorption capacity (q_s) was evaluated as 3.96 mg g^{-1} . The mean sorption energy (E) was found to be $31.62 \text{ kJ mol}^{-1}$ for the adsorption of fluoride on the adsorbent. The E value ranges from 1.0 to 8.0 kJ mol^{-1} denotes physical adsorption and value more than 8.0 kJ mol^{-1} denotes chemical adsorption. The value of E, which is $31.62 \text{ kJ mol}^{-1}$ suggests that the mechanism for the adsorption of fluoride on the adsorbent is a chemical adsorption.

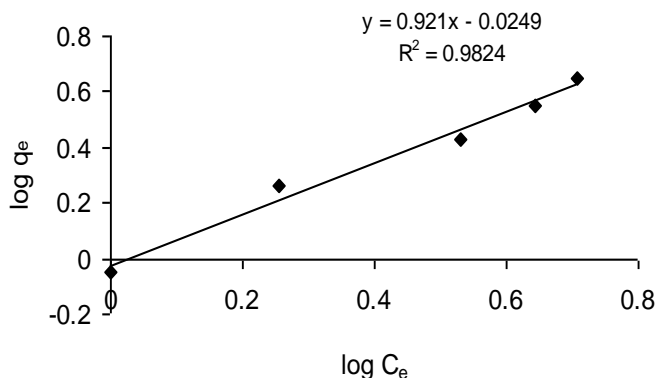


Fig.9 Linearized Freundlich isotherm (initial fluoride concentration = 100 mg/l, equilibrium contact time = 1 h, Temperature = 35°C)

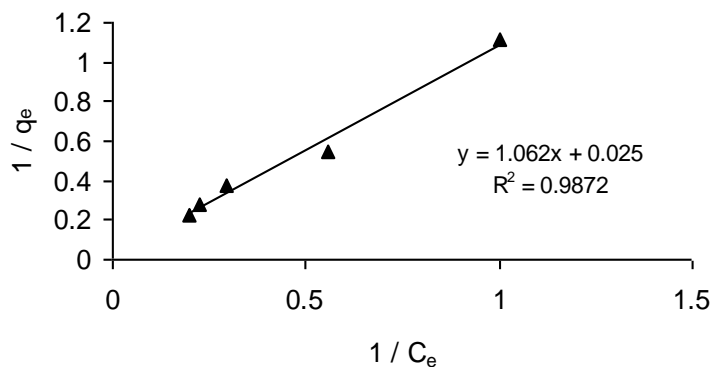


Fig.10 Langmuir isotherm (initial fluoride concentration = 100 mg/l, equilibrium contact time = 1 h, Temperature = 35°C)

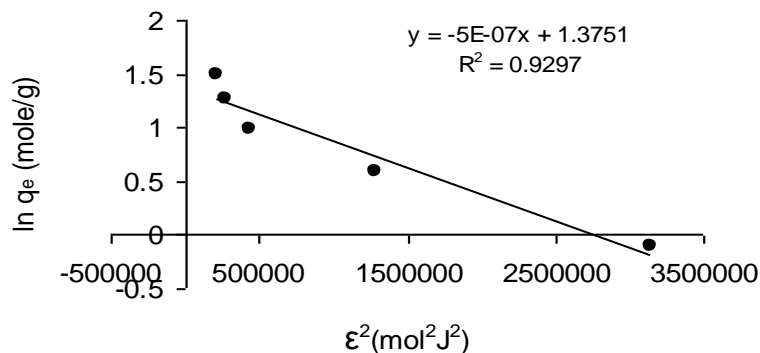


Fig. 11 R-D isotherm (initial fluoride concentration = 100 mg/l, equilibrium contact time = 1 h, Temperature = 35^oC)

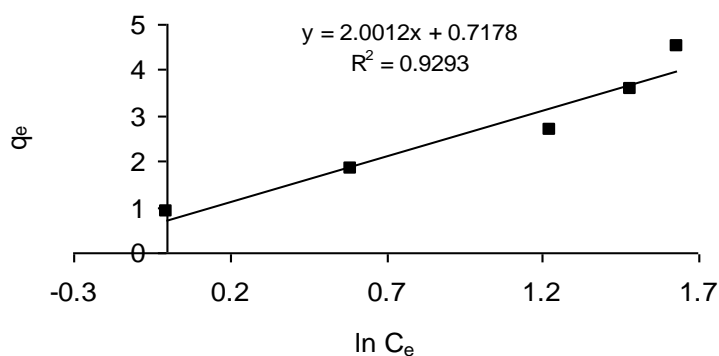


Fig. 12 Temkin isotherm (initial fluoride concentration = 100 mg L⁻¹, equilibrium contact time = 1 h, Temperature = 35^oC)

Adsorption kinetics of fluoride: The adsorption kinetics was studied with initial fluoride concentrations of 100 mg L⁻¹ to 500 mg L⁻¹. The kinetic analysis of the adsorption data is based on reaction kinetics of pseudo-first-order and pseudo-second-order mechanisms. The uptake of fluoride on untreated adsorbent occurred rapidly and reached equilibrium within 60 min. The kinetics of adsorption was analyzed by using the Lagergren equation as shown below:

$$\ln (q_e - q_t) = \ln q_e - K_1 t \quad (19)$$

where q_e and q_t are the amounts of fluoride (mg g⁻¹) on Aloe Barbadensis at equilibrium and at time 't', respectively, and K_1 (min⁻¹) is the first-order rate constant. A plot of $\ln (q_e - q_t)$ against time (t) should yield a straight line (Fig.13), and the rate constant K_1 was evaluated from the slope and was found to be 4.0×10^{-4} , 2.0×10^{-4} , 2.0×10^{-4} , 1.0×10^{-4} and 0.9×10^{-4} min⁻¹ for the concentration of fluoride ion, 100, 200, 300, 400 and 500 mg/l respectively. The values of K_1 were found to decrease with increase in the initial concentration of fluoride from 100 to 500 mgL⁻¹.

For the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$, the integrated form of above equation becomes:

$$1/(q_e - q_t) = (1/q_e) + k_2 t \quad (20)$$

which is the integrated rate law for a pseudo-second-order reaction.

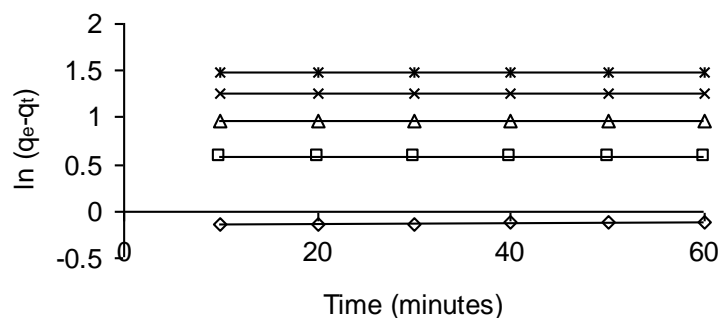
$$t/q_t = (1/k_2 q_e^2) + (1/q_e) t \quad (21)$$

where k_2 (g mg⁻¹ min⁻¹) is the equilibrium rate constant of second-order sorption. The applicability of a particular rate expression for the fluoride-AB system was evaluated from the goodness of data fit and regression coefficient value (R^2). In the present situation the Lagergren first-order equation gives a higher

regression value ($R^2 > 0.9917$) than the second-order equation ($R^2 > 0.8826$), showing adherence to the pseudo-first-order rate law.

Table-2

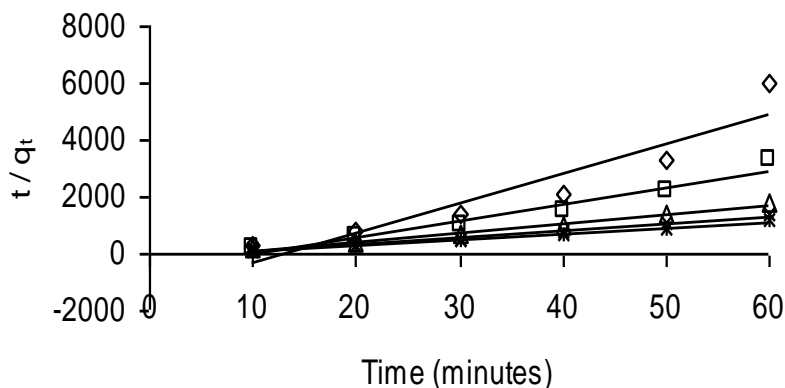
Freundlich Constants	k_f (mg g^{-1}) = 0.94	$1/n = 0.921$	$R^2 = 0.9824$
Langmuir Constants	q_m (mg g^{-1}) = 40	B (mg L^{-1}) = 0.0235	$R^2 = 0.9872$
Dubnina-Radushkevich Constants	q_s (mg g^{-1}) = 3.96	$R_L = 0.2985$	$R^2 = 0.9297$
Temkin Constants	K_T (mg L^{-1}) = 0.2774	E (kJ mol^{-1}) = 31.62	$R^2 = 0.9293$
		$B_1 = 2.0$	



◇ 100 mg/l □ 200 mg/l △ 300 mg/l × 400 mg/l * 500 mg/l

Dose: 10mg, Conc. = 100 mg L⁻¹, Speed = 120 rpm, Speed = 120 rpm

Fig. 13 Lagergren equation plot



◇ 100 mg/l □ 200 mg/l △ 300 mg/l × 400 mg/l * 500 mg/l

Fig.14 Second-order rate equation plot

FT-IR studies : The foregoing results suggested that the adsorption of fluoride ions onto AB may be a complex one involving both physisorptions and chemisorptions. To get a better insight into the nature of the mechanism, the FT-IR spectra of the adsorbents before and after adsorption were recorded and representative spectra are given in fig.15 a and b.

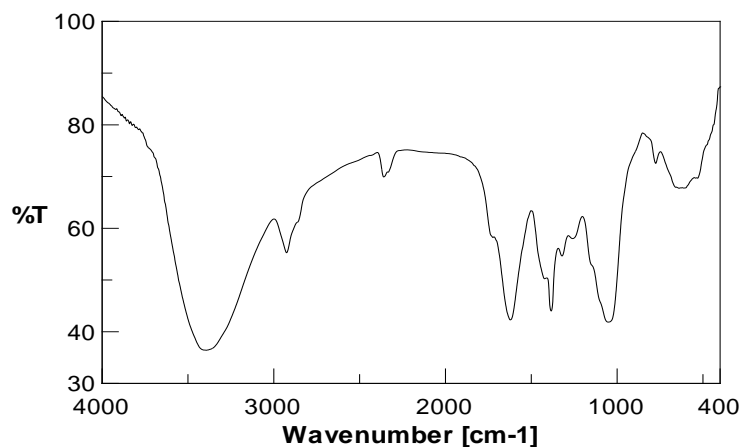


Fig.15 a. FT-IR spectra of AB before adsorption of fluoride ions

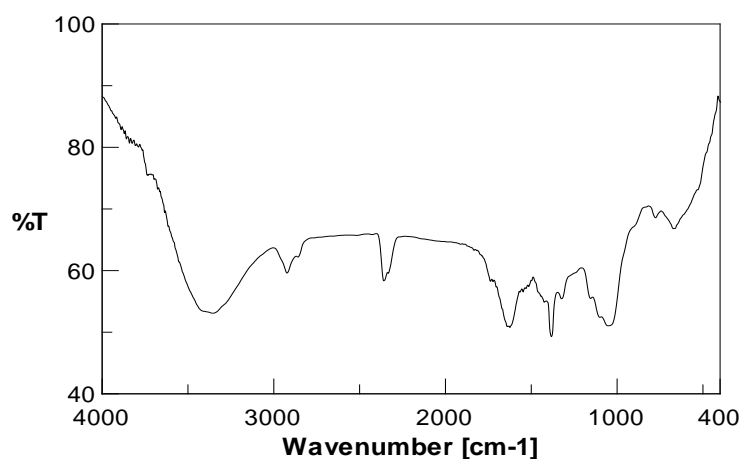


Fig 15 b. FT-IR spectra of AB after adsorption of fluoride ions

APPLICATIONS

This method can be applied for removal of fluoride ions in presence of foreign ions in contaminated water. Among the foreign ions, nitrate showed positive effect while carbonate, sulphate, chloride and phosphate showed negative effect on fluoride removal by AB powder. This might be due to a change in pH and also because of competition between the competing ions and fluoride ions for the active site of AB powder.

CONCLUSIONS

The removal of fluoride ions is maximum for the adsorbent dosage of AB is $10\text{mg } 50\text{mL}^{-1}$. The fluoride adsorption is maximum at 60 min for the adsorbent. The adsorption of F^- ion is maximum in the shaking speed of 120 rpm. The presence of interfering ions such as nitrate showed positive effect while carbonate, sulphate, chloride and phosphate showed negative effect for the adsorbent. In the $\text{pH} = 7$, the F adsorption is maximum for AB. The optimum initial Fconcentration for B adsorbent is $1\text{mg } 50\text{mL}^{-1}$.

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