

Adsorptive Decontamination of Water Containing Fluoride using Waste Material

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Manuscript Received online: (Date), Accepted: ... (Date), Published online (Date)

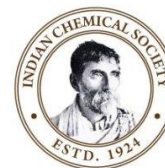
Abstract: In the present study, batch experiments were performed with broken earthen tea cups which are found abundantly as waste material at the road side tea shops. Since these tea cups are made of mud or clay, they do not have any adverse effect of environment. Further, by using these broken tea cups, solid disposal problems are minimized. Several process parameters such as particle size (0.1-0.62 mm), contact time (0.5-10 h), pH (4.5-8.5), adsorbent dose (10-60 gL⁻¹), stirring speed (30-150 rpm), and initial fluoride concentrations (2-12 mgL⁻¹) were varied during batch study and found that the said waste material had good fluoride removal efficiency. At pH 4.5, initial fluoride concentration of 10 mgL⁻¹, particle size of 0.10 mm, adsorbent dose of 50 gL⁻¹ and stirred speed of 120 rpm clay adsorbent showed 75% fluoride removal. Characterizations of the adsorbent were done. Adsorption kinetics was also studied to know the behavior and mechanism of adsorption and Pseudo 2nd order model was found to fit best. Adsorption equilibrium data were fitted reasonably well to Langmuir isotherm model. Regeneration study showed that the adsorbent could be used even after four cycles of operation.

Keywords: Fluoride, Groundwater, Adsorption, Waste Material, Kinetics.

Introduction

The precursor of fluoride compound is fluorine, the lightest member of halogen group. It is the 13th most plentiful element of the earth and is found about 300 mg/kg of earth's shell¹. The contamination of fluoride in groundwater is caused from two sources - natural and anthropogenic sources. Natural sources are mainly associated with the dissolution of fluoride containing minerals into ground water under certain geochemical condition^{2,3}. Besides natural sources, fluoride

is contributed by anthropogenic sources like burning of coal, extraction of aluminium, use of phosphate fertilizers etc. The permissible level of fluoride in drinking water is 1.5 mgL⁻¹ according to World Health Organization^{4,5}. Fluoride is helpful to human health in the range of 0.5 mgL⁻¹ to 1.5 mgL⁻¹ fluoride concentrations⁶. Lesser concentration (<0.5 mgL⁻¹) is responsible for dental fluorosis while, upper concentration (>1.5 mgL⁻¹) leads to skeletal fluorosis and other problems⁷. It may also interfere with DNA synthesis and



metabolisms of carbohydrates, proteins, lipids, vitamins and minerals⁸. The ground water of several continents like Asia, Africa, Australia and South America are badly affected by fluoride. Most fluoride affected states in India include Assam, Andhra Pradesh, Karnataka, Jharkhand, Gujarat and Rajasthan⁹. Therefore, the finding of suitable low-cost material for fluoride removal is very much essential from social, economic and environmental aspects.

Several fluoride removal techniques, such as adsorption, ion-exchange, coagulation-precipitation, and membrane process were attempted to eliminate extra fluoride from contaminated groundwater both at community and household levels^{10,11}. Among those techniques, adsorption has been found to be very efficient due to its simple technical nature and low-cost environment. For the past few years, several adsorbents were performed to investigate their efficiency in the defluoridation process. These adsorbents are laterite soil¹², fly ash¹³, ultrafine tea powder¹⁴, fired clay pots^{15,16}, shale¹⁷, saw dust¹⁸ and other related materials. Because of ease of availability and high effectiveness, low-cost naturally occurring adsorbents have the prospects of great success in the field of fluoride abatement. Therefore, development of naturally available novel low-cost adsorbent is still a major challenge. In the present study, a comprehensive study including kinetic study, equilibrium study, and regeneration study was done for removal of fluoride using naturally available waste material.

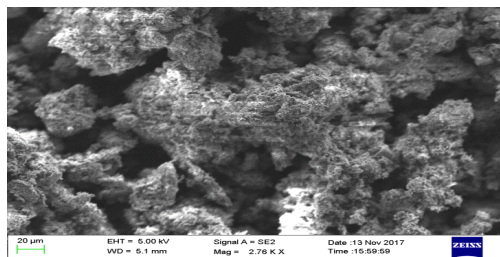
Earthen tea pot, made of mud or clay, is mainly found in road side tea shops. After taking tea, people throw these tea pots to the bin and these spent tea pots are considered as waste material. The main components of earthen tea pots include silica, iron, alumina, calcium oxide, magnesium oxide and other metal oxide¹⁴⁻¹⁶. They do not have any adverse

effect in the environment. Therefore, the present study aims at removal of fluoride from its aqueous solution using such waste material and thereby, the solid disposal problem is also being alleviated. A comparative study was made to assess their efficacies in removal of fluoride by varying different operating parameters like pH, initial fluoride concentration, particle size, dose of adsorbent, contact time and stirrer speed during batch study. The equilibrium and kinetic studies were done for the proper judgement of the mechanism. Regeneration experiments of the adsorbents were carried out to assess the possibility for further use of the spent adsorbent.

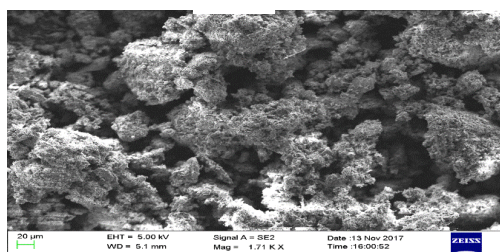
Results and discussion

Characterization of adsorbent

SEM images of earthen tea pots (raw and treated) are shown in Fig. 1(a) and (b). The surface structure of the adsorbent is very much rough and uneven, and small pores are found on the surface. Adsorptive removal increases when there are larger number of pores/adhering sites as present on the surface of adsorbent^{19,20}. During EDX analysis, lines of spectrums (peak) have been obtained which represent the energy (in terms of photons) of each element. The elemental compositions of earthen tea pots are presented in Fig. 2. Treated earthen tea pots contain silica, alumina, iron oxide, calcium oxide, magnesium oxide, sulphur trioxide, phosphorous and other metal oxides as shown in Table 1. The adsorptive capability of earthen pot may be due to the presence of alumina, iron oxide and silica components in the adsorbent.



(a)



(b)

Fig. 1. SEM images: (a) raw tea pot, (b) treated tea pot

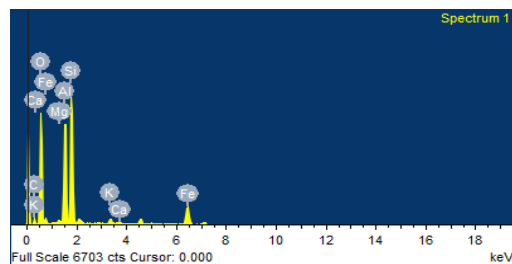


Fig. 2. EDX image of raw tea pot

Fourier transform infrared spectroscopy (FTIR) study has been done in order to investigate the presence of various functional groups in the adsorbent for both before and after adsorption. Fig. 3 shows FTIR peaks of earthen tea pots (before and after adsorption) at different wave numbers. The peaks at wave numbers of 720 cm^{-1} , 1215 cm^{-1} , 1440 cm^{-1} and 3550 cm^{-1} for earthen tea pots before adsorption confirmed the presence of C-H bond, C-O-C bond, -CH₂ bond and O-H bond respectively, and after adsorption, the peaks at

wave numbers of 690 cm^{-1} , 1250 cm^{-1} , 1730 cm^{-1} and 2240 cm^{-1} confirmed the presence of C-H bond, C-O-C bond, -COO bond and O=C=O bond respectively.

Table 1. EDX analysis of earthen tea pots

Compound	Weight (%)
SiO ₂	45.02
Fe ₂ O ₃	32.70
Al ₂ O ₃	12.82
P ₂ O ₅	2.55
CaO	2.26
K ₂ O	1.81
TiO ₂	1.49
MgO	0.80
SO ₃	0.55

Cai *et al.*¹⁴ carried out the FTIR peak studies for ultrafine tea powder as adsorbent. They observed strong peak for tea powder adsorbent at 3630 cm^{-1} , 2350 cm^{-1} before adsorption related to the stretching vibrations of O-H bond and O=C=O bond, whereas peak value at 1240 cm^{-1} and 1650 cm^{-1} related to the vibration of C-O-C bond and -C=O bond after adsorption.

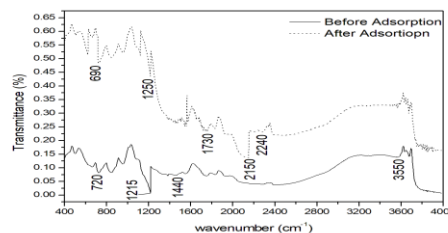
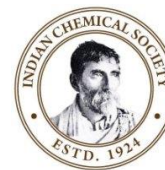


Fig. 3. FTIR image of earthen tea pot

(before and after adsorption)



Optimization of operating variables for removal of fluoride

Effect of particle size

The particle size has been varied to see its effect on fluoride removal efficiency of earthen tea pot during batch study. The results are presented in Fig. 4(a). It has been seen that removal of fluoride decreases with increase in particle size. At low particle size, surface area of an adsorbent is enhanced; since adsorption is a surface phenomenon, removal efficiency increases with decreasing particle sizes. Similar observation was reported by Iriel *et al.*¹² while carrying out removal of fluoride from groundwater using laterite soil. Maximum removal efficiency ($75.63\% \pm 0.84$) was obtained at optimum value of particle size 0.10 mm.

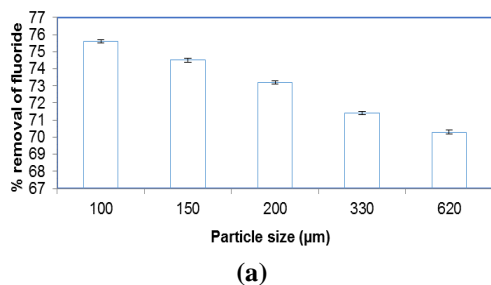


Fig. 4. (a) Particle size variation of fluoride removal using earthen tea pots

Effect of contact time

The effect of contact time on fluoride removal efficiency of earthen tea pot has been investigated next. The results at various contact time are shown in Fig. 4(b). It has been seen that fluoride removal increases with increase in contact time. This is obvious since more the contact time more will be adsorption. However, this will be true before equilibrium is reached. Maximum removal ($74.56\% \pm 0.92$) was achieved at optimum value of contact time 10 h.

However, the removal at 9 h ($73.94\% \pm 0.86$) and 10 h ($74.56\% \pm 0.92$) are almost same. Therefore, optimum value of contact time is 10 h. Kofa *et al.*¹⁵ reported the similar trend.

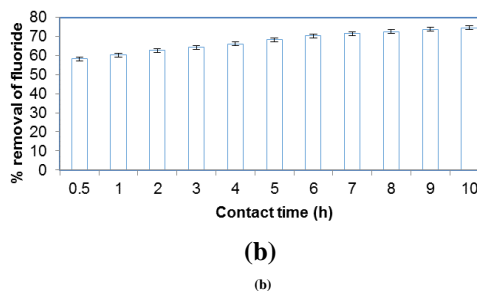


Fig. 4. (b) Contact time variation of fluoride removal using earthen tea pots

Effect of pH

The removal efficiency has been investigated at various pH. The results at different pH are presented in Fig. 4(c). The removal efficiency of fluoride decreases with increase in pH value. At lower pH, the percentage removal of fluoride is higher because of the positive charges present on the surface of the adsorbent that attracts the negatively charged fluoride ions. Hence, removal efficiency decreases with increase in pH. Maximum removal ($77.48\% \pm 2.04$) was

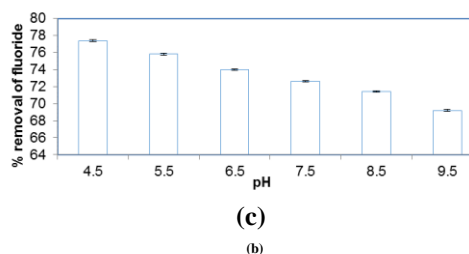
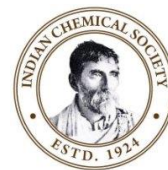


Fig. 4. (c) Variation of pH with fluoride removal using tea pots

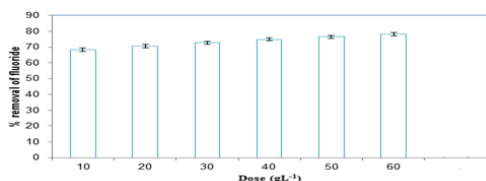
attained at optimum value of pH 4.5. At pH above 7, removal efficiency and capacity of adsorption decreases. It happens because of the competition between hydroxyl ions and fluoride ions towards the active sites²⁰. Araga



*et al.*²³ reported the same variation of fluoride removal with pH.

Effect of adsorbent dose

Effect of adsorbent doses on removal of fluoride has been studied in batch mode. The results with different adsorbent doses are presented in Fig. 4(d). Fluoride removal increases with increasing adsorbent doses up to 50 gL⁻¹. At higher adsorbent doses the removal efficiency increases. Maximum removal (76.52%±1.26) was obtained at optimum value of adsorbent dose 50 gL⁻¹. Above 50 gL⁻¹ adsorbent dose, the removal efficiency of fluoride does not change so much. Motera *et al.*¹⁶ reported similar trend while carrying out the removal of fluoride from aqueous solution using fired clay soil pillared with tea leaf ash components.



(d)

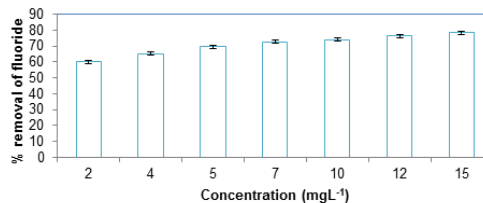
(b)

Fig. 4. (d) Variation of dose with fluoride removal using earthen tea pots

Effect of fluoride concentration

Fig. 4(e) represents the effect of initial concentration on removal of fluoride. Fluoride removal increases with increase in initial fluoride concentration. At higher fluoride concentration (12 mgL⁻¹) the fluoride removal does not change so much. The adsorbent dose used in this study is 50 gL⁻¹. Singh *et al.*¹⁸ reported similar results while carrying fluoride removal from water using saw dust adsorbent. Maximum removal (74.2%±0.56) was achieved at optimum value of initial concentration 10

mgL⁻¹. Biswas *et al.*²¹ also observed similar trend.



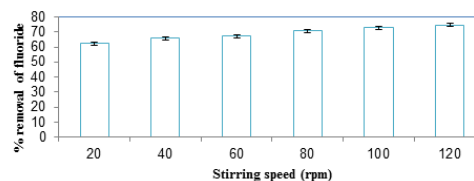
(e)

(b)

Fig. 4. (e) Variation of concentration with fluoride removal using earthen tea pots

Effect of stirring speed

The effect of agitation speed on fluoride removal is shown in Fig. 4(f). It has been seen that fluoride removal increases with increase in stirring speed. At higher stirring speeds the removal efficiency increases. It happens because at higher stirring rate the contact between adsorbent and adsorbate is enhanced^{15,16}. The adsorbent dose used in this study is 50 gL⁻¹. The fluoride removal efficiency decreases when the stirring speed is above 150 rpm. Choudhury *et al.*²² observed the same trend. Maximum removal (75.68%±0.94) was obtained at optimum value of stirred speed 120 rpm.

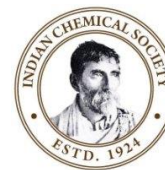


(f)

(b)

Fig. 4. (f) Variation of stirring speed with fluoride removal using earthen tea pots

After treatment at optimum condition (pH: 4.5, adsorbent dose: 50 g/L, initial fluoride concentration: 10 mg/L, particle size: 0.10 mm, contact time: 10 h and stirred speed: 120 rpm), the concentration of fluoride in



groundwater has become 2.5 mg/L (75% removal) which is not within the range of permissible limit. However, by increasing the adsorbent dose, permissible limit may be obtained.

Kinetic study

Kinetic study has been done at optimum condition as obtained from parametric study which is as follows: pH 5.5, adsorbent dose: 50 gL⁻¹, contact time: 10 h and shaking speed: 120 rpm. Kinetic parameters have been evaluated by fitting the kinetic data to the kinetic models. Volume of the solution and temperature has been maintained 200 mL and 25°C. The variation of removal efficiency with time at various initial fluoride concentration has been shown in Fig. 5. It is seen that removal efficiency of fluoride increases with the increase of fluoride concentrations^{15,16,21}. The experimental data have been fitted to various adsorption kinetic models viz., Pseudo 1st order and Pseudo 2nd order models shown in Fig. 6(a) and 6(b). From the results it has been observed that the exploratory data were suitable to fit into the Pseudo 2nd order model over 1st order model shown in Table 2. This happens due to chemisorption of adsorbate molecules. In chemisorption process, the chemical reaction between adsorbent and adsorbate molecules is taken place and chemical bonds are generated on adsorbent surface. These bonds are very strong and effective. Thus, the experimental data are fitted well to Pseudo 2nd order kinetic model. The coefficient values were measured to be sufficient in Pseudo 2nd order kinetic model for tea pots adsorbent (0.982). The calculated q_e values and different kinetic parameters of both Pseudo 1st and 2nd order models using tea pots adsorbent were obtained. It has been shown that experimental and calculated q_e values were very much closed.

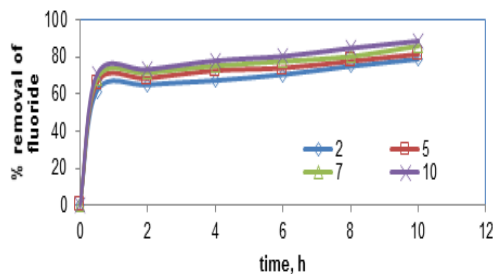
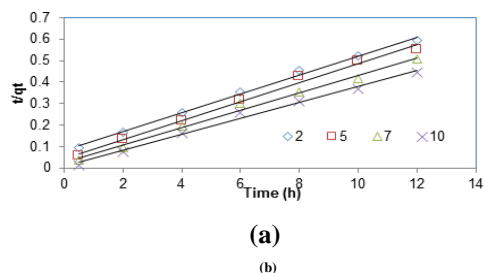
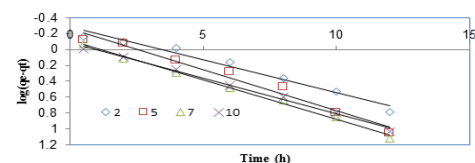


Fig. 5. Kinetic study at different initial fluoride concentration (mgL⁻¹) using tea pots (pH: 5.5, adsorbent dose: 50 gL⁻¹, temperature: 25°C and stirred speed: 120 rpm).



(a)

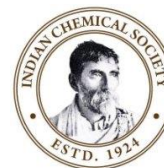


(b)

Fig. 6. Kinetic models. (a) Pseudo 1st order model, (b) Pseudo 2nd order model

Equilibrium study

Adsorption equilibrium study has been done at different fluoride concentrations at pH 5.5, adsorbent dose of 50 gL⁻¹, contact time 10 h, shaking speed 120 rpm and at 25°C. The linearized plot of both Langmuir and Freundlich isotherm models are shown in Fig. 7(a) and (b) to analyze the removal of fluoride from groundwater using earthen tea pots. The values of equilibrium parameters of two isotherm models are shown in Table 3. The experimental equilibrium data are fitted reasonably well to Langmuir isotherm model. The values of adsorption capacity Q_0 and Langmuir constant



b are 0.512 mg/g and 0.0524 for earthen tea pot adsorbent. The correlation coefficient ($R^2 = 0.994$) value for Langmuir model is found to be better than that obtained for Freundlich isotherm model ($R^2 = 0.985$). Kofa *et al.*¹⁵ observed similar results while carrying fluoride removal from water by adsorption onto fired clay pots.

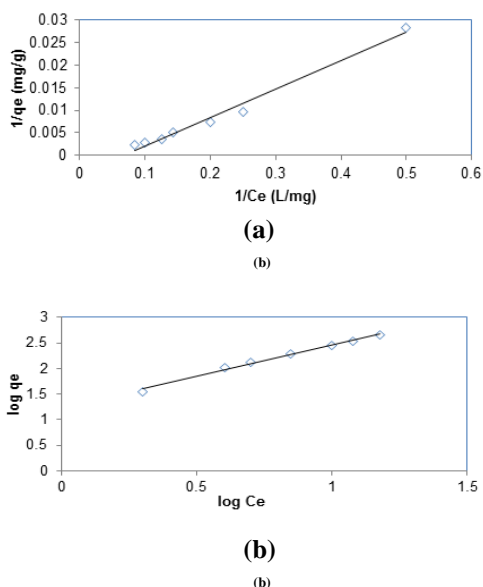


Fig. 7. Adsorption Isotherm models:

(a) Langmuir Isotherm, (b) Freundlich Isotherm

Regeneration study

Regeneration of an adsorbent is of prime importance; as it affirms feasibility of the use of same adsorbent. Desorption capacity of the adsorbent has been checked by treating the already exhausted adsorbent with (M/10) NaOH solution. Further this treated adsorbent has been contacted with the fluoride solution (10 mgL^{-1}). The removal efficiency of regenerated adsorbent has been found to be low. The removal of fluoride decreases from 75% to 68% for usage of adsorbent from first to fourth cycle respectively.

Table 2. Kinetic parameters on removal of fluoride using earthen tea pots

Initial fluoride concentration (mg/L)	Exp. Value q_e (mg/g)	Pseudo order model k_1 (min ⁻¹)	q_e (mg/g)	1 st R^2	Pseudo order model k_2 (g/mg/min)	q_e (mg/g)	2 nd R^2
2	0.256	0.223	1.662	0.915	0.031	0.843	0.932
5	0.478	0.124	1.478	0.942	0.029	1.42	0.956
7	0.627	0.105	1.305	0.957	0.065	3.57	0.971
10	0.854	0.084	1.084	0.974	0.056	5.68	0.982

The regeneration study of these adsorbent is shown in Fig. 8. Saikia *et al.*²⁰ has been observed same results while carrying fluoride removal from contaminated water using carbonized Ipomoea carnea stem carbon coated with aluminum oxyhydroxide nanoparticles.

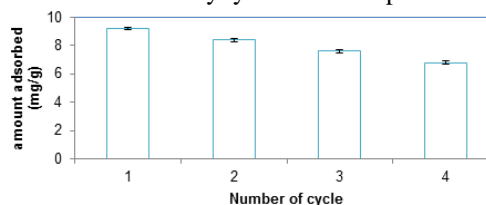
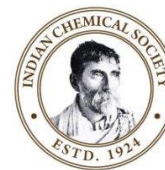


Fig. 8. Regeneration study of earthen tea pot

Experimental Collection, preparation and characterization of adsorbent

Earthen Tea pots were collected from nearby road side tea shops of Durgapur, West Burdwan, West Bengal, India (latitude $23^\circ 52' 04''$ N and



longitude 87° 31' 19'' E). It was then washed with distilled water and dried in hot air oven at 80°C for 1 h. It was crushed, grinded and sieved into different particle sizes (0.1-0.62 mm). Various characterization methods like Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDX) and Fourier Transform Infrared Spectroscopy (FTIR) were employed to study the physico-chemical nature of the earthen tea pots adsorbent before and after treatment. To prepare the fluoride treated earthen tea pots adsorbent, the following protocol was followed: Stock solution of fluoride (1000 mL of 10 mgL⁻¹) was prepared by dissolving requisite amount of sodium fluoride (AR Grade, Merck Ltd.) in distilled water. Weight of adsorbent was 6 g in 200 mL. The solution was then stirred in a rotary shaker for different contact time (0.5-10 h) and after the stirring the flasks was left for 0.5 h. The slurry was filtered using Whatman 42 filter paper and the fluoride treated solid adsorbent was used for characterization study.

During SEM study, surface of sample is scanned with the aid of electron beams. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. Scanning Electron Microscopy (SEM; Model E1010, HITACHI, SIGMA ZEISS) was used to study the morphological analysis of earthen tea pot adsorbent. Energy Dispersive X-ray Spectroscopy is used to give a better analysis of the engaged elements in any sample. EDX analysis was done to study the elemental composition of the used adsorbent.

Table 3. Langmuir and Freundlich isotherm parameters for fluoride removal

Adsorbent	Langmuir Model	Isotherm		Freundlich Model	
	Q ₀ (mg/g)	b (L/mg)	R ²	K _F (mg/g)	n R ²
Earthen tea pots	0.512	0.0524	0.994	25.36	0.821 0.985

The Fourier Transform Infrared Spectroscopy accumulates high-spectral-resolution data over range of wide spectra to identify the different functional groups as present in the sample. FTIR analysis of earthen tea pot adsorbent before and after adsorption of fluoride was done using the FTIR spectrometer (Model No: SHIMADZU IRPRESTIGE-21).

Optimization of operating parameters for removal of fluoride

To evaluate the effect of different operating parameters on fluoride removal, particle sizes (0.1-0.620 mm), contact time (0.5-10 h), pH (4.5-8.5), adsorbent dose (10-60 gL⁻¹), stirrer speed (30-150 rpm) and initial fluoride concentration (2-12 mgL⁻¹) were varied individually during adsorption using earthen tea pots in batch study. Three sets of experiments were performed to assess repeatability of data. To each of the flasks, 200 mL fluoride solution was added with 6 g amount of adsorbent. The solution was then stirred in a rotary shaker for different contact time. The solution was left for 0.5 h. It was then filtered using filter paper. The final fluoride concentration was measured by Thermo Scientific Orion ion meter (model no: Orion star A214 pH/ ISE meter) with the help of ion selective electrode after standard



calibration by adding TISAB-III buffer solution.

Kinetic study

To assess the mechanism of adsorption, kinetic study is essential. The fluoride solutions with different initial concentrations (2-10 mgL⁻¹) were contacted with earthen tea pot separately for 10 h. Adsorbent dose, pH, stirring speed and temperature were maintained at 50 gL⁻¹, 5.5, 120 rpm and 25°C respectively. Samples were collected after a definite time interval. The samples were then filtered and final concentrations of fluoride were measured. Two kinetic models viz., Pseudo 1st order and Pseudo 2nd order kinetic models were employed to analyze kinetic data.

Equilibrium study

Batch experiments were conducted at optimum condition as determined during parametric study which was follows: pH 5.5, adsorbent dose: 50 gL⁻¹, stirrer speed: 120 rpm, contact time: 10 h and at 25°C. Initial concentrations of fluoride solution were varied in the range of (2-10 mgL⁻¹). Samples were collected after 10 h and analyzed for residual fluoride concentration. The adsorption isotherm is an equation relating the amount of solute adsorbed on the surface of solid and the equilibrium solute concentration in solution at a given temperature. In the present experiment the data for the removal of fluoride using earthen tea pots were fitted to the most commonly used adsorption isotherm namely, Langmuir and Freundlich adsorption isotherm models¹⁵⁻¹⁷.

Regeneration study

Regeneration study is performed to check the repetitive use of any adsorbent. To examine the reusability of the adsorbents, several cycles of operation were carried out. Initial fluoride concentration of 10 mgL⁻¹ was used to check adsorption cap-ability of

regenerated spent adsorbent. Desorption study was performed by contacting the already exhausted adsorbent with (M/10) NaOH solution (up to four cycles).

Conclusions

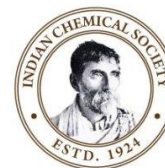
Removal of fluoride from groundwater using low cost waste material like earthen tea pots has been carried out in the present study. Several process parameters such as particle size, contact time, pH, adsorbent dose, stirring speed, and initial fluoride concentration have been varied and it has been found that the said material has good fluoride removal efficiency. At pH 4.5, initial fluoride concentration of 10 mgL⁻¹, particle sizes of 0.10 mm, adsorbent dose of 50 gL⁻¹, and shaking speed of 120 rpm earthen tea pots adsorbent showed 75% fluoride removal efficiency in batch mode. Adsorption kinetics has been studied in an exhaustive way to know the behavior and mechanism of adsorption. The adsorption data has been applied to both Pseudo 2nd order and 1st order kinetic models and found that Pseudo 2nd order model fitted best. Adsorbent equilibrium study was done and fitted reasonably well to Langmuir isotherm model. Adsorbent is fairly feasible as even after four cycles of operation. Therefore, it can be concluded that naturally available low-cost waste material like earthen tea pots have the potential in fluoride removal from contaminated groundwater.

Conflict of interest

The authors declare that they do not have any conflict of interest.

Acknowledgements

The authors would like to extend warmest gratitude to the Department of Chemical Engineering, National Institute of Technology, Durgapur and Director of CSIR-CMERI (Central Mechanical Engineering Research



Institute), Durgapur, India for supporting to carry this research work.

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