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DEFLUORIDATION OF WATER BY A BIOMASS: SHOREA ROBUSTA

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ABSTRACT

This paper focused on search of biomass which can effectively remove fluoride from drinking water. Sal (Shorea robusta) leaf powder was found to reduce fluoride. The biosorbent of 0.3 mm and 1.0 mm sizes of Sal leaf was prepared by standard sieve. Batch experiments done to see the defluoridation properties from synthetic solution of 5 ppm to study the influence of parameters pH, adsorbent dose, contact time and initial fluoride concentration on adsorption efficiency. The specific uptake of Sal powder 0.3mm and 1mm increases from 0.0195 mg/g to 0.158mg/g and 0.0135mg/g to 0.127mg/g respectively with the increase in initial concentration of from 1.6 mg/l to 5mg/l. The maximum specific uptake obtained from Langmuir isotherm is found to be 1.28 mg/g by Sal powder (0.3mm). The values of adsorption capacity (Kf) and intensity of adsorption (1/n ~ 0.84 <1) indicate Freundlich as a favourable sorption model. The fluoride removal efficiency of Sal leaf powder of 0.3mm and 1mm particle size was about 63.6% and 25.8% respectively observed at pH 7.5, keeping the concentration 5mg F/L and at 1g dosage of biosorbent in 50ml solution and 98.6% at 3g dose of Sal powder(0.3mm). Adsorbent size of 0.3 mm was more efficient than the 1.0 mm size. Characterization of biosorbents, before and after adsorption was studied by Fourier Transform Infrared (FTIR) and Scanning Electron Micrograph (SEM) coupled with EDX to get a well understanding of the mechanism of adsorption.

Keywords: Shorea Robusta, Defluoridation, Biosorbent, Batch Experiment

I. INTRODUCTION

Persistence of fluoride in drinking water need urgent attention as it can cause dental and skeletal fluorosis when present at levels above 1.5 mg/l (WHO 2008). Strong electonegativity of fluoride ion is attracted towards positively charged calcium ion in teeth and bone which results in dental, skeletal and no skeletal forms of fluorosis i.e. high fluoride ingestion, in children as well as adults. Various diseases such as osteoporosis, arthritis, brittle bones, cancer, infertility, brain damage, Alzheimer syndrome, and thyroid disorder can attack human body on excessive intake of fluoride [1]. The problem of fluoride contamination of groundwater which is a major source of drinking water is exasperating day by day. Defluoridation of water is the only practicable option to get rid from excess fluoride. Defluoridation techniques can be broadly classified in to four categories; Adsorption technique, Ion-exchange technique, Precipitation technique, and other techniques, which include electro chemical defluoridation and Reverse Osmosis. Adsorption is the most simple, appealing, easy handling, efficient, environmental friendly and economic amongst the above technique. Further, use of plant materials as an adsorbent is seeking researcher's interest due to their abundant availability and low cost. Besides this the presence of chemical functional groups such as hydroxyl, carbonyl, carboxyl, sulfhydryl, theioether, sulfonate, amine amide, imidazole, phosphonate, and phosphodiester on the biosorbent surface contribute to biosorption[8][9][10]. Several literatures are available on the removal of fluoride from water using various biodsorbents like mosambi fruit peel powder[2], *Phyllanthus emblica*[3], *Citrus limonum*(lemon) leaf[4], rice husk ash, neem leaf[5], peepal leaf , khair leaf[6], tamarind fruit shell[7] etc. In the current work the potential of locally available Sal leaf for the removal of fluoride from water has been explored. The characterization of these biosorbents has been done by using scanning electron microscope (SEM) coupled with EDX and Fourier Transform Infrared (FTIR). The effects of solution pH, adsorbent dose, agitation time and initial fluoride concentration on the removal of fluoride have been done in batch reactor configuration and equilibrium parameters have been estimated.

II. MATERIALS AND METHODS

2.1 Preparation of Bio-Sorbents

Mature and dry fallen leaves of Sal were collected from BIT campus. Semi dried leaves were dried in hot air oven in the range of 80-100ºC till they became crisp. Dried leaves were crushed by hand and ground by a mechanical grinder. After grinding sieved through standard sieve to obtain particle of sizes up to 1mm and 0.3mm. Thus, the prepared bio-sorbent of Sal leaf power were used for sequestration of fluoride from aqueous solution. Elemental analysis of untreated Sal leaf powder is shown in Table1.

2.2 Characterization of Adsorbents

Various characteristics like pore size, functional group of prepared adsorbents before and after adsorption were studies by using SEM coupled with EDX and FTIR techniques.

III. BIOSORPTION EXPERIMENT

Biosorption capacity of biosorbent (Sal leaf powder) of different sizes was determined by using batch sorption experiments adopting independent flask method. In this method, each flask containing adsorbent and fluoride mixture. The stock solution of 100 mg/L fluoride was prepared by dissolving 221 mg of anhydrous NaF in one liter of distilled water. Test solution of 5 mg/L fluoride concentration was prepared from stock solution. This concentration was selected as upto this concentration of fluoride found in drinking water. A series of 100 ml Stoppard flask containing 50 ml solution and adsorbent, were shaken in a horizontal shaker, to study the various parameters at room temperature. At the end of desired contact time, the conical flasks were removed from the shaker, and allowed to stand for 5 min for the adsorbent to settle. Subsequently, samples were filtered using Whatman No. 42 filter paper and 5 ml filtrate from each batch were analysed for residual fluoride concentration by SPADNS photometric method, described in the Standard Methods of Examination of Water and Wastewater [11] at 570 nm using the UV–Vis spectrophotometer. To get the optimum process conditions for biosorbent the effect of process variable such as pH, adsorbent dose, time and initial fluoride concentration on the removal of fluoride was studied. The conditions used for these experiments are shown through Table 2. The effect of pH on fluoride was studied by adjusting the pH of test solution using 0.1 N HCl or 0.1 N NaOH on fixed quantity of adsorbent.

% adsorption (A) of fluoride was calculated as using following formula:

$$\%A = [(C_i - C_f) \times 100]/C_i$$

Where Ci and Cf are the initial and residual concentration at equilibrium (ppm)

Table1. Elemental Analysis and Physical Properties of Bio-sorbent

S. No.	Name	Elemental analysis (%)				Physical properties	
		C	H	N	S	Particle size (μm)	Wt.(mg)
1.	Sal Leaf	46.37	7.096	0.387	0.283	0.3	5.8260

Table2. Ranges of Operating Parameters for pH, Adsorbent Dose, Time and Initial Fluoride Concentration for the Removal of Fluoride

Objective of experiment	**Values of variable parameters**	**Values of fixed parameters for verifying optimum conditions**
To study the effect of pH on fluoride removal	Solution pH: 6.5, 7, 7.5, 8, 8.5	AD: 1g/50ml; IFC: 5 ppm; Temp.: Room temp.;Time: 1hr., Particle size:0.3mm, 1mm
To study the effect of time on fluoride removal	Time: 10, 15, 20, 25, 30 min.	AD: 1g/50mL; IFC:5 ppm; Temp.: Room temp.; Solution pH:7.5, Particle size:0.3mm,1mm
To study the effect of adsorbent dose on fluoride removal	AD: 0.5, 1, 1.5, 2, 2.5, 3 g/50mL.	IFC: 5ppm; Temp.: Room temp. ; time: 1 hr.; Solution pH 7.5
To study the effect of initial fluoride ion concentration on fluoride removal	IFC: 1.6, 2, 2.5, 3, 3.5, 4, 5 ppm	AD:1g/50mL;Time.:1hr; Solution pH:7.5; Temp: Room temp.

IV. RESULTS AND DISCUSSION

4.1 Characterization of the Bio-Sorbents

4.1.2 FTIR Analysis

The FTIR spectra of Sal leaf powder were obtained to understand the nature of the functional groups present in it. FTIR spectra (Fig.1) displayed a number of peaks, indicating the complex nature of the adsorbent. The broad and strong band ranging from 3000 to 3600cm^{-1} indicates the overlapping of Si–OH (silanol), R–OH (hydroxyl) and –NH2 (amine) stretching vibrations. The band at 2921 to 2929 cm^{-1} was assigned to C-H stretching [12].

The peaks located at 1735 and 1719 $cm^{-1}$are characteristic of carbonyl group stretching from carboxylic acid or ester group. The band at 1617 to 1637 cm^{-1} was assigned to the asymmetric stretching of –COO- in ionic carboxylic group .The band at 1384 cm^{-1} was assigned to symmetric –COO- stretching in pectin [13]. The band at 1021 to 1054 cm^{-1} was assigned to C-OH stretching in alcohols. Among these functional groups, carboxylic and hydroxyl groups had great significance in the removal of fluoride ions. On comparing the IR spectra it was found that there are significant changes in the fresh and exhausted biomass spectra. The identification of absorption spectrum shows the range, type, vibration and functional groups of the biomass. FT-IR study of fresh and exhausted Sal powder (Figure 1(a), (b)) shows shifting of functional group plotted in Table 3. major differences in the region 500 - 1600 cm^{-1} range is assigned to $-NH_2$ groups in different parts of the biomass. Frequency ranges of biomass indicating bonding of fluoride with the amines groups of biomass. These stretching in functional groups are responsible for fluoride adsorption.

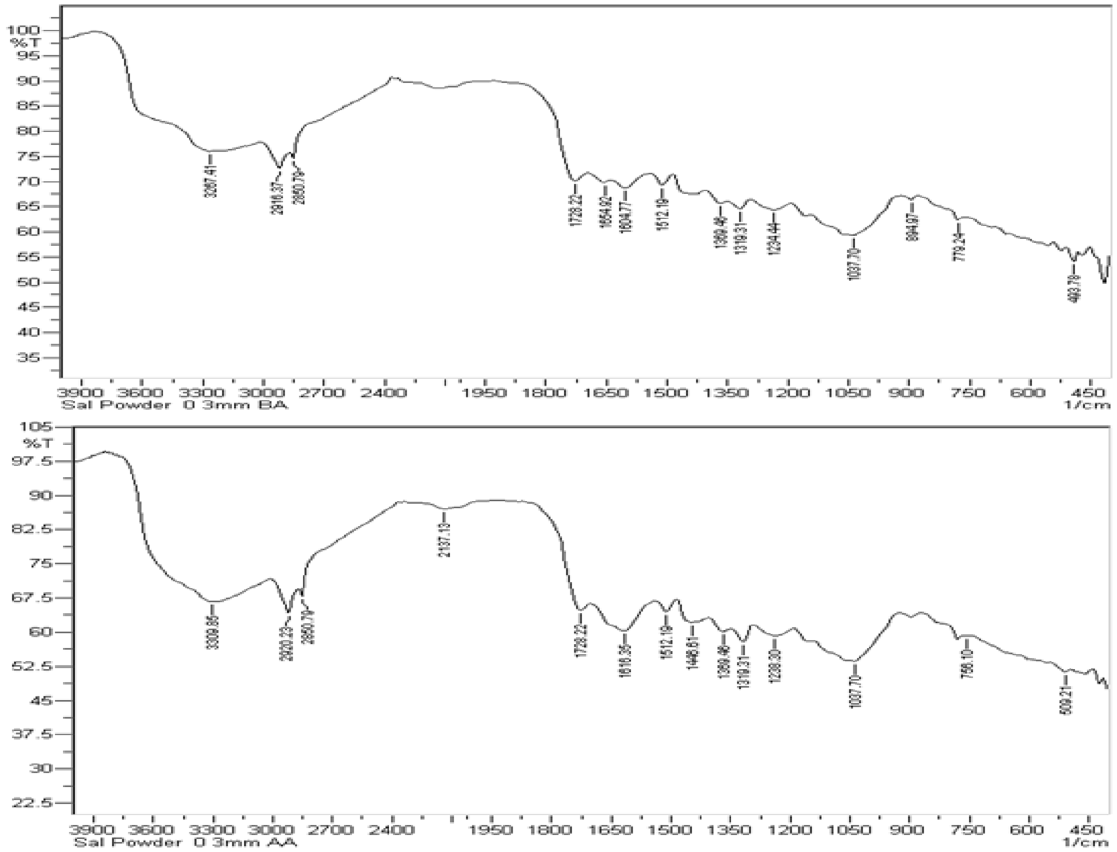

Figure 1(a) FT-IR spectra of fresh (BA) & exhausted (AA) Sal Powder (0.3mm)

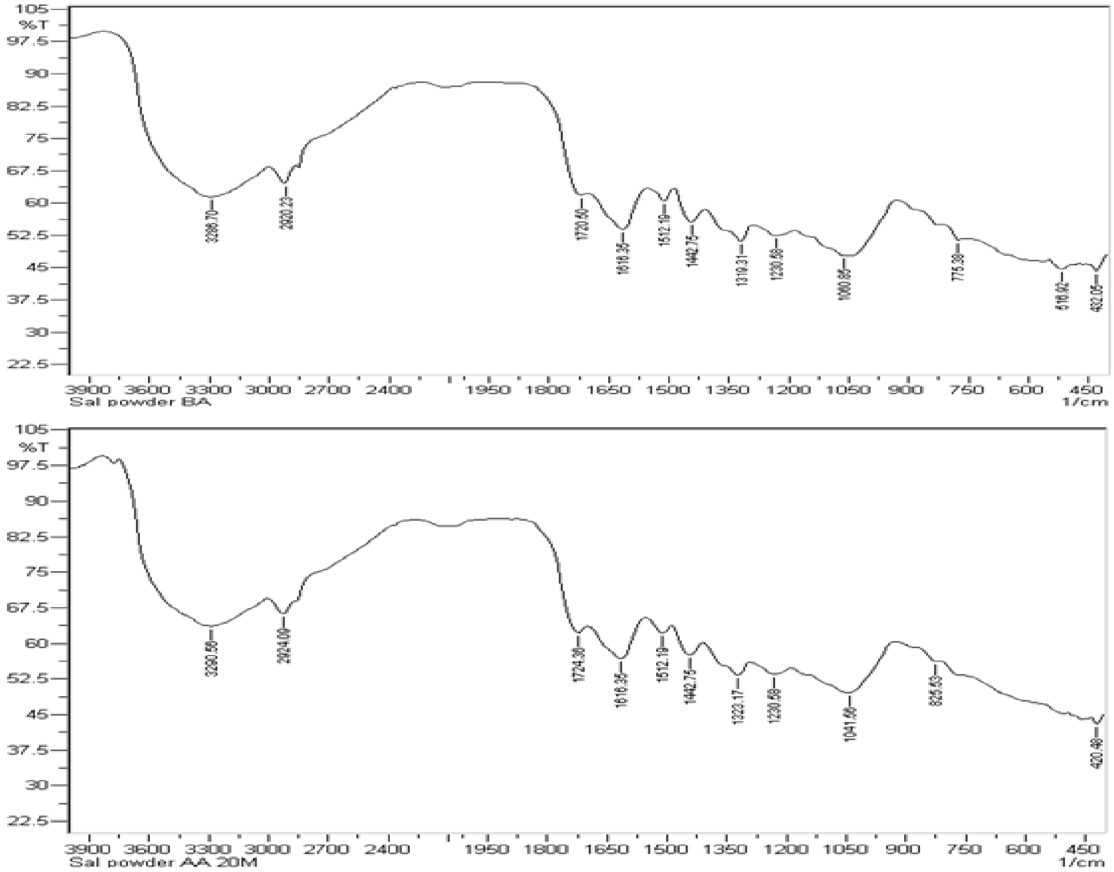

Figure 1(b) FT-IR Spectra of Fresh (BA) & Exhausted (AA) Sal Powder (1mm)

Table 3 FT-IR Analysis

Functional groups		**Sal (1mm)**	**Sal (0.3mm)**
OH free alcohols	**BA**	3286.70	3267.41
	AA	3290.56	3309.85
C-H stretching	**BA**	2920.23	2916.37
	AA	2924.09	2920.23
C=O stretching vibration (non-ionic)	**BA**	1720.50	1728.22
	AA	1724.36	1728.22
COO- asymmetric stretching (ionic)	**BA**	_	_
	AA	_	_
C=O deformation stretching	**BA**	1230.58	1234.44
	AA	1230.58	1233.30
C-OH stretching in alcohols	**BA**	_	1037.70
	AA	1041.56	1037.70
-NH_2 groups	**BA**	516.92	_
	AA	_	509.21

4.1.2 BA (Before Adsorption), AA (After Adsorption)

Besides these peak 1604.77 shifted to 1616.35 for Sal 0.3mm and peak 1319.31 to 1323.17, 1000.85 to 1041.56 for Sal 1mm.

4.2 SEM Analysis Coupled with X-ray Energy Dispersive Spectroscopy (EDX)

SEM and EDX are useful tools for evaluating the elemental characteristics of the adsorbents and these two techniques have been widely used in adsorption studies especially in determining the adsorption mechanism [22][23][24][25]. The SEM images and EDX spectra for Sal leaf powder before and after fluoride adsorption are shown in Fig. 2 and Fig. 3 for Sal powder 0.3mm and 1mm respectively. The SEM images (at 800× magnification) show the rough surface and porosity of Sal powder, conditions which might favour the adsorption of fluoride ions. The EDX spectrum for Sal powder (0.3mm) before adsorption (Fig. 2) showed some peaks for Ca, K, C, P, O and Si species. The EDX analysis gave different results and this analysis could be used to confirm the attachment of F ions onto Sal surface. The presence of fluoride on Sal surface is represented by the peak F (Fig. 2b). After F adsorption the intensity of Ca, Si, C, P decreased (Table 4). Similarly, the EDX spectrum for Sal powder (1mm) before adsorption (Fig. 3) showed some peaks for Ca, P, O and Si species. The EDX analysis gave different results and this analysis could be used to confirm the attachment of F ions onto Sal surface. The presence of fluoride on Sal surface is represented by the peak F (Fig. 3b). After F adsorption the intensity of Ca, Si, and P decreased (Table 5). The decreased intensity of these cations peaks and the appearance of F peak as revealed by EDX spectra indicated that ion-exchange involved during adsorption process. This kind of observation has also been reported in our previous studies [26][27].

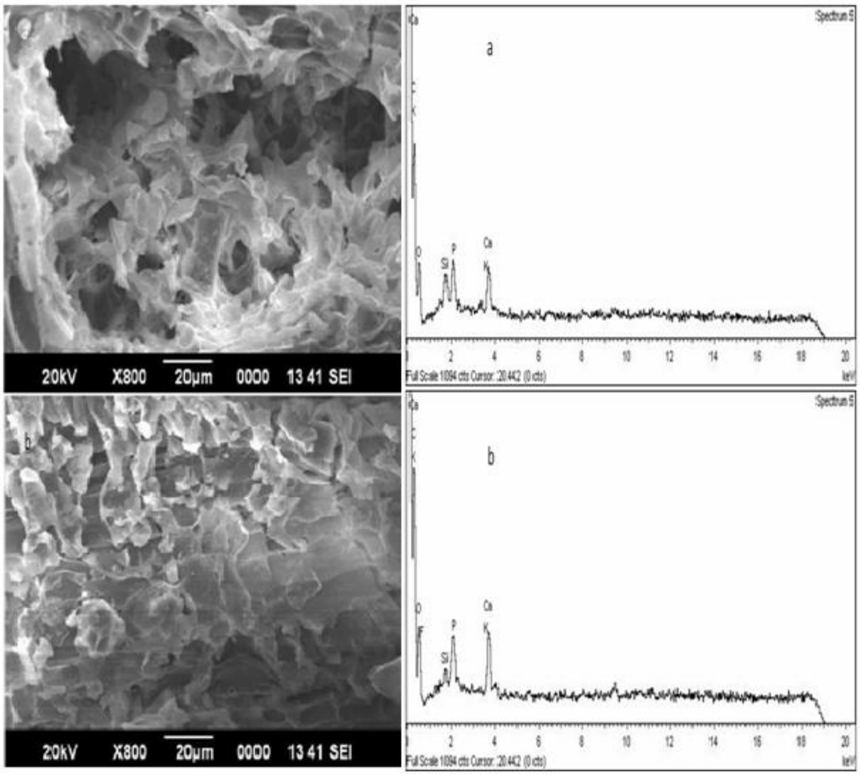

Figure 2. SEM Images at 800× Magnification and EDX Spectra of Sal Powder (0.3mm) Before (a) and After (b) Fluoride Adsorption

Elemental Composition of the Sal Powder

(0.3mm) Before and After Fluoride Treatment as Revealed by EDX Analysis- (Table 4)

Analysis Before Adsorption								
Elements		Ca	O	K	Si	P	C	Total
Weight %		12.93	67.17	2.14	7.47	5.48	7.34	100
Analysis After Adsorption								
Elements	F	Ca	O	K	Si	P	C	Total
Weight %	11.27	12.90	61.37	0.26	2.80	4.77	6.59	100

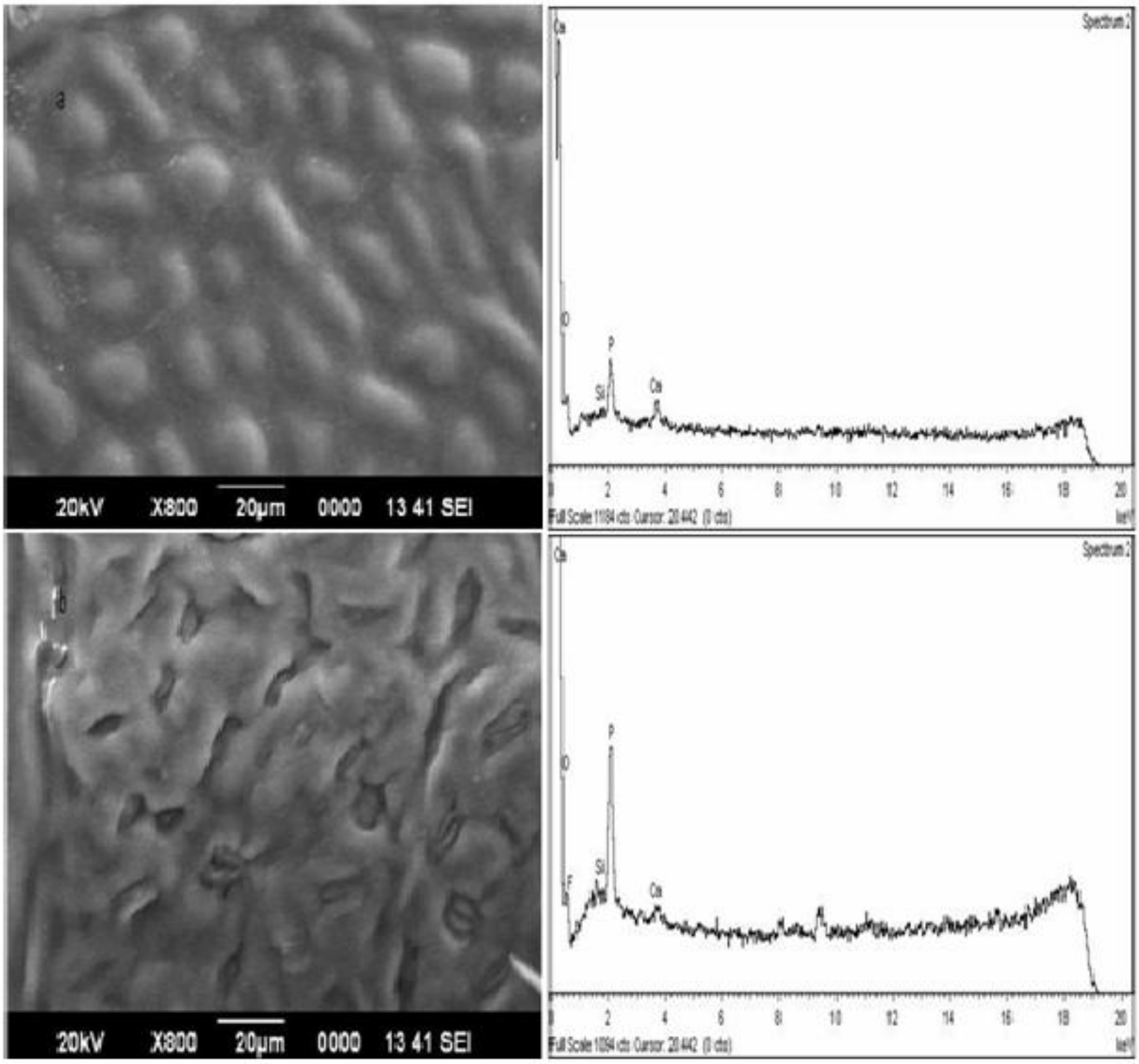

Figure 3. SEM Images at 800× Magnification and EDX Spectra of Sal Powder (1mm) Before (a) and After (b) Fluoride Adsorption.

Elemental Composition of the Sal powder (1mm) Before and After Fluoride Treatment as Revealed by EDX Analysis- (Table 5)

Analysis Before Adsorption						
Elements		Ca	O	P	Si	Total
Weight %		10.49	77.79	8.19	3.52	100
Analysis After Adsorption						
Elements	F	Ca	O	P	Si	Total
Weight %	36.41	2.21	47.03	12.17	2.18	100

V. EXPERIMENTAL RESULT

5.1 Effect of pH

The effect of pH on fluoride adsorption was examined at 5 mg/L of F ion concentration and sorbent concentration of 1.0 g in 50ml. From figure 4, it seems that at lower pH, % removals of fluoride for Sal leaf powder are less, which increases gradually with the increase in solution pH, reach maximum at around pH 7.5 and then decreases. It is evident that fluoride removal reached a maximum of 63.6% and 25.8% by particle size 0.3mm and 1mm respectively of Sal powder at pH 7.5. The solution pH alters the surface charge of the adsorbent and solution phase chemistry of the adsorbate. Thus, shows significant effects on the removal of adsorbate from water. The mechanism of adsorption of fluoride ion on adsorbent surface is explained by following reactions.

$$HF \leftrightarrow H^+ + F^- \qquad (1)$$

$$\equiv POH + H^+ \leftrightarrow POH_2^+ \qquad (2)$$

$$\equiv POH + OH^- \leftrightarrow PO^- + H_2O \quad (3)$$

$$\equiv POH_2^+ + F^- \leftrightarrow PF + H_2O \quad (4)$$

$$\equiv POH + F^- \leftrightarrow PF + OH^- \qquad (5)$$

Where POH, POH_2^+ and PO^- are the neutral, protonated and deprotonated sites on leaf powder and PF is the active site of fluoride complex.

At lower pH, the fluoride ions which exist as HF weakly ionized and the surface of the sal leaf powder is primarily positive since more protonation takes place with the adsorbent sites through equation 2, thus the removal of fluoride is less. But with the increase in solution pH the dissociation of the HF increases and the positive charge density of adsorbent also gradually decreases. However, above pH 7.5 the increase in solution pH converts the surface of the Sal leaf powder adsorbent as predominantly negatively charged through equation 3, where fluoride also exists as negatively charged fluoride ion. Thus, the repulsive force between leaf powder adsorbent and the fluoride reduces chemisorption of fluoride at pH greater then 7.5 as a result the % removal of fluoride decreases which results maximum removal of fluoride at pH 7.5.

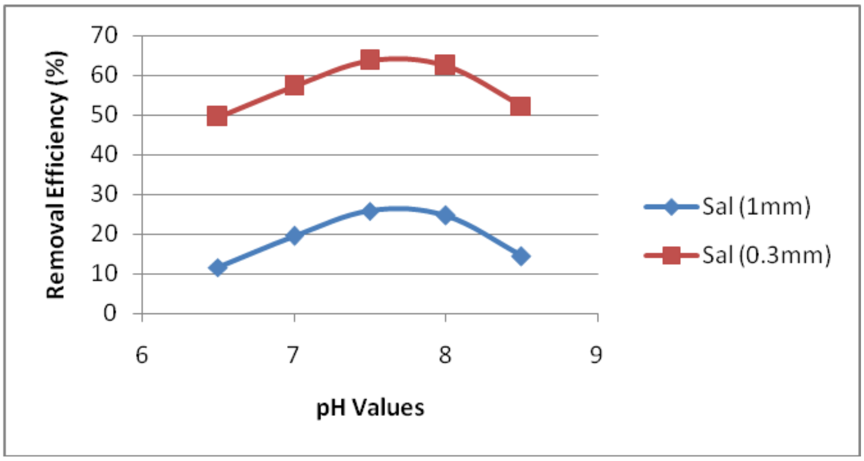

Fig .4 Effects of pH on Fluoride Sorption onto Sal Leaf Powder.

5.2 Effect of Time

The effect of contact time on the removal of fluoride from drinking water is shown in figure5. It was noted that the percentage fluoride removal was increasing with time and attained almost an equilibrium condition in about 25 minutes at which, the rate of adsorption of solute was equal to the rate of desorption. The decrease in the rate of removal of fluoride, particularly towards the end indicates a possible monolayer of fluoride on the outer interface of the adsorbent and pore (intra-particle) diffusion on to the inner surface of the adsorbent particles through the film due to continuous agitation maintained during the experiments. At the contact time of 25 min the % fluoride removal by Sal powder (0.3 mm and 1.0 mm) increases from 46.4% to 57.8% and 14.4% to 40.0%, respectively.

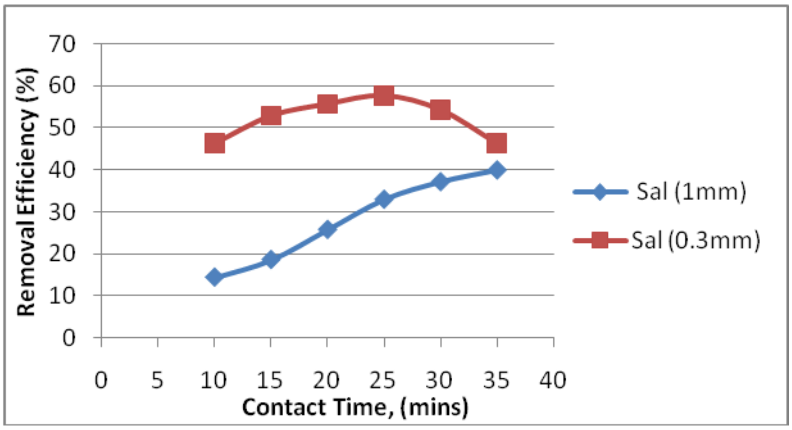

Fig .5 Effects of Contact Time on Fluoride Sorption onto Sal Leaf Powder.

5.3 Effect of Adsorbent Dose

The percentage removal of F ion was studied varying the adsorbent dosage from 0.5- 3 g/L at optimum conditions of extraction viz., initial concentration of fluoride ion : 5.0 mg/L; contact time : 60 min at 30°C. Figure 6 shows fluoride uptake by both Sal powder of 0.3mm and 1mm particle size at different dose of 0.5 g to 3.0 g. It is also found that as particle size increase there was decrease in defluoridation capacity. It is observed that fluoride removal by 0.3 mm and 1 mm sizes adsorbent increases from 42.2% to 98.6% and 51.0 to 87.2%

respectively at 0.5 g to 3.0 g of Sal powder/ 50 ml. Hence, the maximum removal of fluoride ions can be obtained by using 3 g in 50 ml. Initially the increase in % removal with increase in adsorbent dose increases as the removal of adsorbate depends on the number of available active sites, which increases with increase in adsorbent dose.

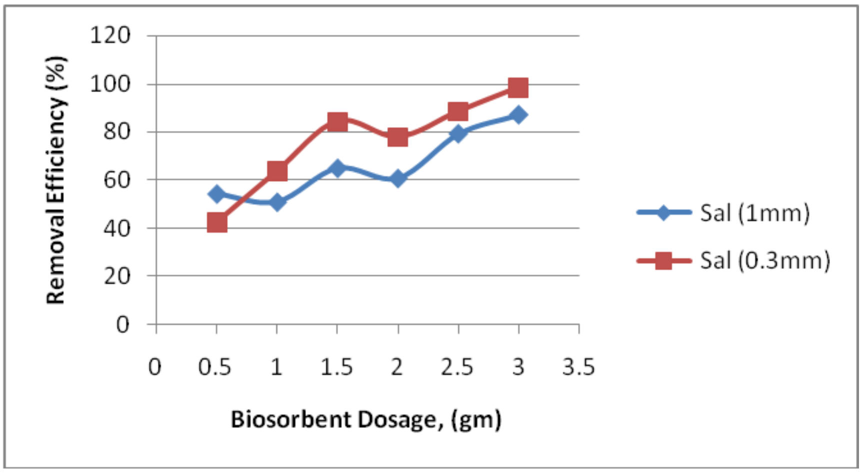

Fig .6 Effects of Adsorbent Dosage on Fluoride Sorption onto Sal Leaf Powder.

5.4 Effect of Initial Concentration

The effect of initial concentration of fluoride on the extent of specific uptake of fluoride in terms of amount of the fluoride adsorbed on the Sal powder adsorbent was studied with a fixed dose of adsorbent: 1.0 g in 50 ml solution, constant agitation time of 60 minutes and at temperature: 28-30°C, by varying the initial concentration of fluoride between 1.6 - 5.0 mg/L. The specific uptake of fluoride by adsorbent for the removal of fluoride from water is shown in figure7. From the figure it is clear that the specific uptake of Sal powder 0.3mm and 1mm increases from 0.0195 mg/g to 0.158mg/g and 0.0135mg/g to 0.127mg/g respectively with the increase in initial concentration of from 1.6 mg/l to 5mg/l. With the increase in initial concentration of fluoride the driving force for transport from bulk to the surface of adsorbent increases, which results more adsorption of fluoride per unit mass of adsorbent.

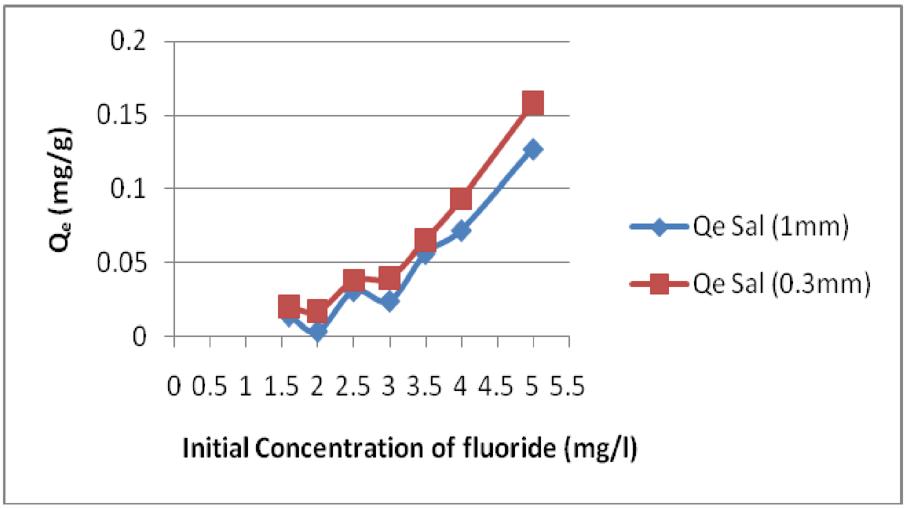

Fig .7 Effects of Initial Fluoride Concentration on its Sorption onto Sal Leaf Powder

5.5 Equilibrium Study for Fluoride Removal

Freundlich and Langmuir isotherm models were tested to find out suitable model equation for predicting equilibrium adsorption of fluoride from water. Linear forms of these isotherms and values of isotherm parameters are described in Table 6 and 7 respectively.

Table 6. Equilibrium Models

Freundlich model	$\log Qe = \log Kf + 1/n \log Ce$
Langmuir model	$1/Q_e = 1/Q_o + 1/bCeQ_o$

Langmuir adsorption isotherm assumes the existence of monolayer coverage of adsorbate on the structurally homogeneous surface of the adsorbent. All the sorption sites are assumed to be identical and energetically equivalent [14]. In equilibrium model given in Table 6 Qe is the metal ion adsorbed at equilibrium (mg/g), Ce is the equilibrium concentration of the metal ion (mg/L) b and Qo are the constants related to the energy of adsorption and the maximum adsorption capacity respectively, [15], [16]. The values of Qo and b were calculated from the intercept and slope of the linear plot of 1/Qe against 1/Ce. The equilibrium data was fully described by the Langmuir adsorption isotherm as shown in figure below.

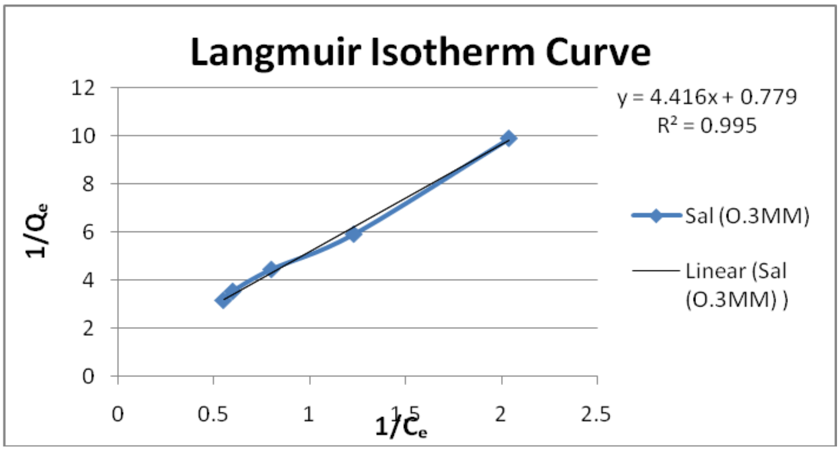

Fig. 8 Langmuir Plot for Removal of Fluoride at 1 g Sal Powder (0.3mm)/100 mL Volume, pH 7, Temp of 30 °C, Contact Time of 60 Min and Varied Initial Concentration of Adsorbate.

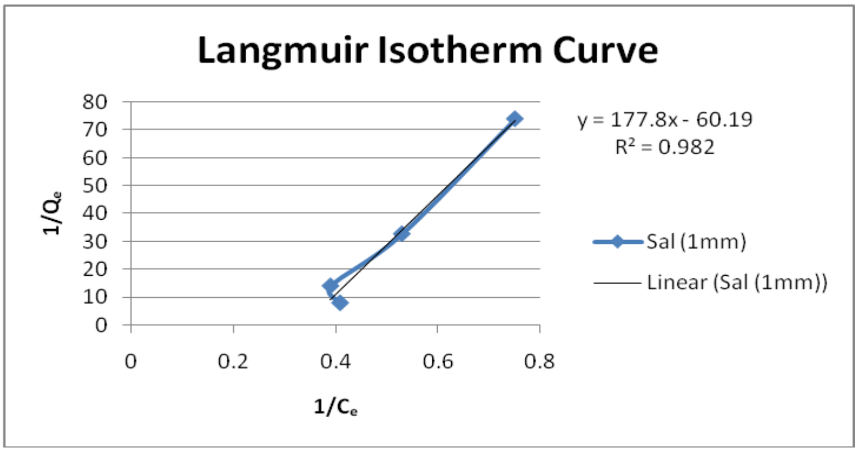

Fig.9 Langmuir Plot for Removal of Fluoride at 1 g Sal Powder (1mm)/50 mL Volume, pH 7, Temp of 30 °C, Contact Time of 60 Min and Varied Initial Concentration of Adsorbate.

A low b value on the Langmuir adsorption parameters indicated the low affinity of the sorbent for the sorbate [17].

Freundlich adsorption isotherm assumes that the adsorption occurs on amorphous surface that is heterogeneous and there is exponential distribution of active sites and their energies [20]. The logarithmic form of the Freundlich adsorption isotherm is expressed in Table 6 above [18, 19] where Qe and Ce have similar meaning to those in the Langmuir adsorption isotherm while Kf and 1/n are the adsorption capacity and adsorption intensity respectively. Kf and 1/n were obtained from the intercept and gradient of the linear plot of logQe against logCe. The Freundlich adsorption parameters are shown in Table 7. The R² value confirmed the fitness of the data to this model [21]. The adsorption intensity parameter (1/n), was within the range 1< n <10 that shows that there was easy separation beneficial biosorption and therefore must be capitalized.

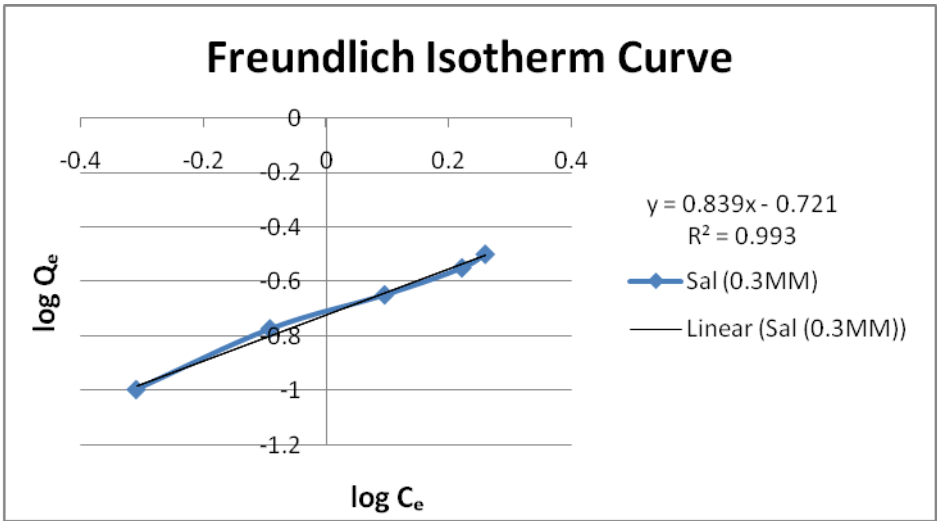

Fig.10 Freundlich Plot for Removal of Fluoride at 1 g Sal Powder (0.3mm)/100 mL Volume, pH 7, Temp of 30 °C, Contact Time of 60 Min and Varied Initial Concentration of Adsorbate

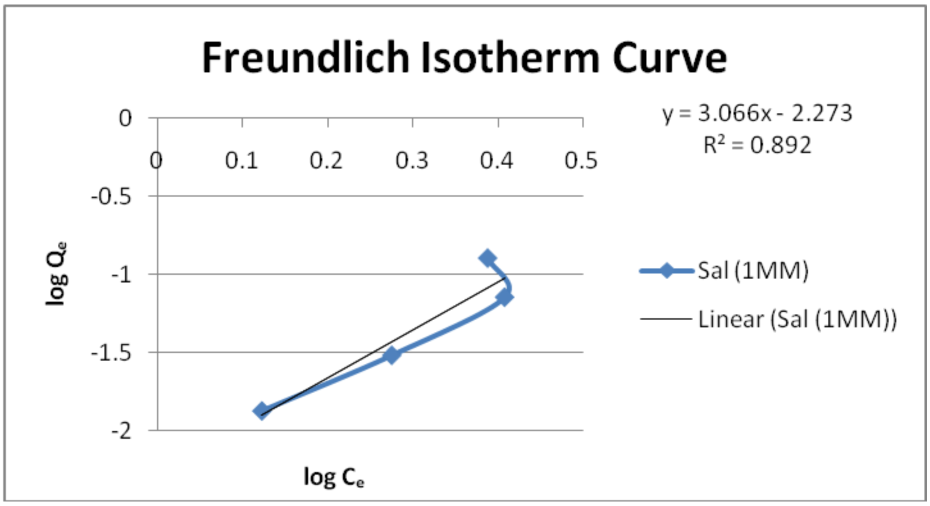

Fig. 11 Freundlich Plot for Removal of Fluoride at 1 g Sal Powder (1mm)/50 mL Volume, pH 7, Temp of 30 °C, Contact Time of 60 Min and Varied Initial Concentration of Adsorbate

Table7: Adsorption Isotherm Parameters for Fluoride Sorption onto Sal Leaf Powder.

Isotherms	**Parameters**	**Values**	
		Sal (0.3mm)	**Sal (1mm)**
Freundlich	R^2	0.993	0.892
	N	1.19	0.326
	1/n	0.84	3.06
	K_f	0.190	0.0053
Langmuir	R^2	0.995	0.982

VI. CONCLUSIONS

Sal leaf powder proved to be an excellent biosorbent for F^- ions from aqueous solution at optimum pH 7.5. Fluoride ions are found to adsorb on the surface of Sal leaf powder. The results suggest that pore diffusion is more important. The specific uptake of Sal powder 0.3mm and 1mm increases from 0.0195 mg/g to 0.158mg/g and 0.0135mg/g to 0.127mg/g respectively with the increase in initial concentration of from 1.6 mg/l to 5mg/l. The maximum specific uptake obtained from Langmuir isotherm is found to be 1.28 mg/g by Sal powder (0.3mm). The values of adsorption capacity (Kf) and intensity of adsorption (1/n ~ 0.84 <1) indicate Freundlich as a favourable sorption model. Results also indicate that the fluoride removal efficiency of Sal leaf powder of 0.3mm and 1mm particle size was about 63.6% and 25.8% respectively observed at pH 7.5, keeping the concentration 5mg F/L and at 1g dosage of biosorbent in 50ml solution and 98.6% at 3g dose of Sal powder(0.3mm). SEM coupled with EDX characterizations of biosorbents show evidence for inner-sphere complications and gives a clear picture of change in morphology before and after fluoride treatment that confirms adsorption of fluoride. FT-IR spectra revealed the presence of O-H, C-H, C=O, C-OH and $-NH_3$ stretching in the adsorbents showed fluoride binding in the different frequency ranges of the biosorbents. This plant biomass is recommended as a suitable and low cost adsorbent to reduce fluoride and 0.3 mm size was more efficient than 1.0 mm.

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