

INTRODUCTION

According to a report of 2013, 1 in every 9 people (approximately 783 million) lack access to safe water. Furthermore, more than 6.8 million people die from a disaster or water-related disease [1]. Fluoride contamination in drinking water due to natural and anthropogenic activities has been recognized as one of the major problems worldwide, imposing serious threat to human health. A maximum of 1.5 mg fluoride is permitted by WHO in 1 L of water [2]. Fluoride concentration in drinking water above 1.5 mg/L may cause serious harm to human health.

More than 200 million people in 29 counties are suffering from fluoride contamination of groundwater. Some of the countries in which fluoride contamination of groundwater have been significant include India, Argentina, and Ethiopia [3,4]. Widespread occurrence of fluoride above the prescribed limit in groundwater meant for human consumption has caused multidimensional health problems, most common being dental fluorosis, and skeletal fluorosis [5,6]. Maximum permissible amount of fluoride by various organizations worldwide has been reported in Table 1 [7].

A report published by Rajiv Gandhi National Drinking Water Mission in 1993 identified 15 states including Delhi as endemic for fluorosis. Table 2 depicts states which are presently endemic for fluorosis [8].

Problem of fluorosis is increasing day by day as fluoride level is gradually getting higher than the prescribed maximum desirable limits. The fluoride level in water in India ranges from 2 to 29 ppm, whereas the permissible level in drinking water according to WHO standard is 1.0-1.5 ppm. In tropical countries, skeletal fluorosis occurs even with drinking low level of fluoride. Epidemiological observation revealed that nutritional status might influence chronic fluoride toxicity.

In India, people are consuming fluoride in water up to 18 mg/L. It is easily absorbed by the body from contaminated drinking water. After absorption, fluoride ion is quickly distributed throughout the body, easily crossing the membranes and going into tissues. It accumulates in body due to the high reactivity of fluoride ion with calcium of teeth and bones. It forms calcium fluorophosphates (fluorapatite) crystal and leaves unbound calcium in the same tissue, which gets calcified and in turn results in stiffness of tissues and joints. This finally leads to skeletal fluorosis in a later stage. That’s why fluoride is called as bone-seeking mineral and bones as sink for fluoride. About 90% of the fluoride retrieved in body is associated with calcified tissues.

Several defluoridation methods proposed and tested worldwide are mostly based on the principles of precipitation [9], adsorption [10-12], and ion exchange [13]. However, the defluoridation methods developed so far lack viability at the end-user level due to one or more reasons such as high-cost technology, limited efficiency, unnoticeable break through, deteriorated water quality, and taboo limitations [14]. These methods involve the use of alum, lime, aluminum sulfate, gypsum-fluorite filters, magnesite, dolomite, poly aluminum chloride, granulated bone media, bone charcoal, and activated carbon, and activated alumina, superphosphate, and tri-calcium phosphate [15,16]. Among above-mentioned methods, adsorption is one of the most economically feasible techniques used for water defluoridation. In this paper, we have studied the adsorption of fluoride in fixed bed column using java plum (Syzygium cumini) as previously in several literatures java plum had been used as biosorbent for several harmful contaminants [17,18]. Here, a fixed bed column has been used specifically because their residence time distribution studies proved them to be a good model for adsorption [18]. This paper basically represents the study of the defluoridation capacity of the java plum seed.



METHODS

Preparation of adsorbent

Java plum (S. cumini) seed was collected from the local market of Roorkee, Hardiwar, and Uttarakhand. After the collection of seed, it was
washed three times from the tap water to remove the dust. Java plum (S. cumini) seed was primarily dried in the sunlight for 2 days and then dried in hot air oven in the range of 80-100°C for 36 hrs. The material is subsequently placed in an airtight container for further use.

**Column adsorption experimental**

This experiment was carried out in a reactor column of SS pipe with an objective to remove fluoride from wastewater. The schematics diagram of the experimental setup was shown in Fig. 1. SS pipe column of various length (Z<sub>1</sub>=20, Z<sub>2</sub>=40, Z<sub>3</sub>=60, Z<sub>4</sub>=80 and Z<sub>5</sub>=100 cm) and 8.5 cm internal diameter were used while the height of reactor was 100 cm and net volume 5.03 L. It was equipped with a total of four equidistant ports (excluding inlet and outlet) of 1.25 cm diameter for collecting liquid sample along the height of reactor. The top and bottom portion were connected with the main column by two flange joints, supported on SS screen (mesh no: 16 BSS, width aperture: 1.00 mm). The reactor is filled with weighted amount of java plum (S. cumini) seeds (biosorbent) having a particle size of 2-4 mm as a fixed-bed absorber. The bed was supported and closed by cotton pad and rubber, respectively, to prevent the flow of adsorbent together with the effluent. Then, the bed was rinsed with distilled water and left overnight to ensure a closely packed arrangement of a particle without voids, channels, or cracks. Synthetic fluoride solution of known concentration (20 mg/L) was fed through a bed of java plum (S. cumini) seeds in up-flow mode to avoid channeling due to gravity and to ensure a uniform distribution of the effluent thought out the column. The experiments were carried out at room temperature. A peristaltic pump was used to control the flow rates (12, 23 and 40 ml/min) and maintained constant during each experiment. Periodic flow rate check carried out by collecting sample at the effluent for a given time and measured using measuring cylinder. A sample of effluent was collected at 1 hr interval and analyzed by spectrophotometric (SPADNS) method for fluoride ion concentration using ultraviolet spectrophotometer (Hach, DR 5000). The volume of treated water was measured at 1 hr interval, and the average flow rate was calculated based on these values because the flow rate becomes unstable as the bed depth is high due to a higher flow resistance. The desired breakthrough concentration (C<sub>b</sub>) was determined at 75% of the initial concentrations (20 mg/L).

**Mathematical modeling**

**Thomas model**

Thomas model was one of the most general and widely used models in the column performance theory. Thomas model was based on the assumption that the process follows Langmuir kinetics of adsorption-desorption with no axial dispersion. The prediction of column adsorption process requires prediction of breakthrough curves for the effluent. The maximum adsorption capacity of an adsorbent is also required in the design of column. The mathematical representation for a fixed bed column with ideal break through curve can be expressed as follows [19,20]:

\[
\frac{C_t}{C_0} = \frac{1}{1 + \exp \left( \frac{q_m \cdot m - C_0 \cdot V}{Q \cdot v} \right)}
\]

The linearized form of the column,

\[
\ln \left( \frac{C_0}{C_t} - 1 \right) = k_m q_t \cdot m \cdot V \cdot \ln \left( \frac{C_t}{C_0} \right)
\]

Where,

- C<sub>0</sub>: Initial solute concentration (mg/L),
- C<sub>t</sub>: Solute concentration at time, t, (mg/L),
- V: Volumetric flow rate (ml/min),
- q<sub>m</sub>: Maximum solid phase concentration of solute (maximum column adsorption capacity) (mg/g),
- k<sub>m</sub>: Thomas rate constant (L/min mg),
- m: Mass of absorbent (g).

k<sub>m</sub> and q<sub>m</sub> can be determined from the slope and the intercept of the linear plot of ln((C<sub>t</sub>/C<sub>0</sub>)-1) versus V, respectively.

**Thomas model was based on the following assumptions:**

i. Langmuir kinetics of the adsorption-desorption;

ii. No axial dispersion which was derived with the assumption that the rate driving force obeys second-order reversible reaction kinetics.

**Table 1: Permissible limit of fluoride in drinking water prescribed by various organizations**

| Organization                              | Maximum permissible limit of fluoride ions |
|-------------------------------------------|-------------------------------------------|
| International standard for drinking water | 0.5                                        |
| U.S. Public Health Standard               | 0.8                                        |
| The committee on public health            | 1.0                                        |
| engineering manual and code of practice,  |                                            |
| Govt. of India                            |                                            |
| Indian Council of Medical Research        | 1.0                                        |
| recommendation                            |                                            |
| ISI recommendation                        | 1.5                                        |
| WHO Guidelines                            | 1.5                                        |
| Bureau of Indian Standard                 | 1.2                                        |

**Table 2: Summarized information on the occurrence of excessive fluoride in ground water in India**

| State             | Number of habitation with excess fluoride | State             | Number of habitation with excess fluoride |
|-------------------|------------------------------------------|-------------------|------------------------------------------|
| Andhra Pradesh    | 7548                                     | Madhya Pradesh    | 201                                      |
| Gujarat           | 2378                                     | Orissa            | 1138                                     |
| Karnataka         | 860                                      | Punjab            | 790                                      |
| Kerala            | 287                                      | Rajasthan         | 16,560                                   |
| Meghalaya         | 33                                       | Tamil Nadu        | 527                                      |
| Haryana           | 334                                      | Uttar Pradesh     | 1072                                     |
| Himachal Pradesh  | 488                                      | West Bengal       | 21                                       |
Table 3: Salient feature of column reactor

| S. No. | Description                              | Value  |
|--------|------------------------------------------|--------|
| 1      | Diameter of reactor (cm)                 | 8.0    |
| 2      | Total height of reactor (cm)             | 100    |
| 3      | Volume of reactor (L)                    | 5.03   |
| 4      | Number of sampling points                | 5.0    |
| 5      | Height of sampling point (cm)            | 100    |
| 6      | Diameter of sampling point (cm)          | 1.25   |
| 7      | Total weight of absorbent (g)            | 2420   |
| 8      | Density of bed (g/ml)                    | 0.7166 |
| 9      | Actual volume of reactor (liters)        | 1.623  |

### RESULTS

#### Fixed-bed design models

The bed depth service time model (BDST), the EBRT model and the Thomas model were selected for this study which was used to predict, optimize and describe the fixed-bed column operation, respectively.

**BDST model**

Fig. 2 showed the BDST plots \((T_b \text{ versus } D)\), which was constructed from Table 4 for the influent fluoride concentration of 20 mg/L and flow rates of 12, 23 and 40 ml/min at 7.5% breakthrough time for 20, 40, 60 and 100 cm bed heights. The coefficients \(N_b\) and \(K\) for the three flow rates were calculated based on equation:

\[
T_b = \frac{N_b D}{C_b V} - \frac{1}{K C_b \ln \left( \frac{C_b}{C_b} \right)}
\]

Where,

- \(T_b\): Service at breakthrough point (h),
- \(N_b\): Bed capacity (mg cm\(^{-2}\)),
- \(D\): Packed-bed column depth (cm),
- \(C_b\): Influent fluoride concentration (mg/L),
- \(C_{Cb}\): Breakthrough fluoride concentration (mg/L),
- \(K\): Adsorption rate constant (L/mgh).

The equation of a straight line on BDST curve could be expressed as

\[
y = ax + b
\]

Where,

- \(y\): Service time,
- \(x\): Bed depth,
- \(a\): Slope,
- \(b\): Ordinate intercept.

The numerical value of the slope \((a) = N_b/C_b V\) and the intercept \((b) = \frac{1}{K C_b \ln \left( \frac{C_b}{C_b} \right)}\).

The adsorbent concentration from exceeding \(C_{br}\) was obtained when \(T_b = 0\), according to the following equation:

\[
D_{min} = \frac{v \ln \left( \frac{C_0}{C_{br}} \right)}{K N_0}
\]

The slope of the line presented by \(y = ax + b\) could be used to predict the performance of the bed if there was change in the initial solute concentration \(C_0\) to a new \(C_{br}\).

Hutcheson proposed that the new slope \(a_2\) and new intercept \(b_2\) could be estimated by equation [25].

\[
a_2 = a_1 \frac{C_{br1}}{C_{br2}}
\]

\[
b_2 = b_1 \frac{\ln \left( \frac{C_{br1}}{C_b} \right)}{\ln \left( \frac{C_{br1}}{C_{br2}} \right)}
\]

McKay stated that if design data were required for a change in volumetric flow rate of solute to the same adsorption system, the new slope with the intercept remaining unchanged could be written as [25].

\[
a_2 = \frac{a_1 Q_1}{Q_2} = \frac{ay_1}{y_2}
\]

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Effect of bed depth

The adsorption of fluoride onto java plum (S. cumini) seeds was presented in the form of breakthrough curves where the concentration ratio $C_t/C_0$ was plotted against time (h). Figs. 3-5 showed the breakthrough curves of fluoride adsorption onto java plum (S. cumini) seeds at different bed depth (20, 40, 60, and 100 cm) and at a constant flow rate of 12, 23 and 40 ml/min. The result indicated that breakthrough volume $V_b$ and breakthrough time $T_b$ increases with increasing bed depth.

The breakthrough time $T_b$ was directly related with $D$ according to the equation

$$T_b = \frac{NaD}{C_0V} \times \frac{1}{KC_0 \ln \left( \frac{C_0}{C_b} - 1 \right)},$$

thus as bed depth increased the breakthrough time also increased which in turn lead to an increase in treated volume ($V_b$). When the bed depth increases, the adsorption performance increases due to increase in adsorption mass which provided greater adsorption site.

The breakthrough curve indicated in Fig. 3 showed that at lower bed depths, the shape of the curve does not show the characteristic S-shape profile produced in ideal adsorption systems, but exclusively for the 100 cm column the shape in approaching S-shape curve.

Increasing bed depth increases the EBRT from 106.03 to 530.04 for 12 ml/min flow rate, and similar trends were also followed with other flow rates.

![Fig. 2: Bed depth service time plot at 7.5% breakthrough in a fixed-bed column at different flow rates](image)

![Fig. 3: Effect of bed depth on breakthrough time at a constant flow rate of 23 ml/min ($C_0=20$ mg/L)](image)

![Fig. 4: Effect of bed depth on breakthrough time at a constant flow rate of 12 ml/min ($C_0=20$ mg/L)](image)

![Fig. 5: Effect of bed depth on breakthrough time at a constant flow rate of 40 ml/min ($C_0=20$ mg/L)](image)

**Table 4: Data of variable bed depth at a fixed flow rate in a fixed-bed column for the removal of 20 mg/L of fluoride by java plum (S. cumini) seeds (Biosorbent)**

| Q flow rate (ml/min cm²) | Bed height (cm) | Bed volume (cm³) | Weight of adsorbent (mg) | EBRT (min) | $V_b$ (L) | $T_b$ (h) | Adsorbent exhaustion rate (g/L) |
|-------------------------|----------------|-----------------|--------------------------|------------|-----------|---------|-------------------------------|
| 12 ml/min 0.1886 (ml/min cm²) | 20  | 1272.34  | 483.4  | 106.03  | 2.947  | 4  | 163.97 |
| 40 | 2544.68  | 969.1  | 212.01  | 13.465  | 18  | 71.97 |
| 60 | 3817.02  | 1451.9  | 318.0  | 20.818  | 29.33  | 69.74 |
| 100 | 6361.7  | 2419.9  | 530.14  | 29.042  | 42  | 83.324 |
| 23 ml/min 0.3615 (ml/min cm²) | 20 | 1272.34  | 483.4  | 55.319  | 2.031  | 1.5  | 238.01 |
| 40  | 2544.68  | 969.1  | 110.639  | 7.988  | 6  | 121.32 |
| 60  | 3817.02  | 1451.9  | 165.957  | 19.264  | 14  | 75.36 |
| 100 | 6361.7  | 2419.9  | 276.595  | 22.5  | 78.09 |
| 40 ml/min 0.6287 (ml/min cm²) | 20 | 1272.34  | 483.4  | 31.080  | 1.074  | 0.45  | 45.09 |
| 40  | 2544.68  | 969.1  | 63.617  | 7.574  | 3.17  | 127.95 |
| 60  | 3817.02  | 1451.9  | 152.68  | 11.676  | 4.83  | 124.09 |
| 100 | 6361.7  | 2419.9  | 159.04  | 15.644  | 6.5  | 154.72 |

EBRT: Empty bed residence time, S. cumini: Syzygium cumini
Effect of flow rate

Fig. 6 shows that the influence of rate on the time of breakthrough at fixed-bed depth of 100 cm. An increase in flow rate from 23 to 40 ml/min reduces the volume of treated at the breakthrough and therefore decreased the service time of the bed. As could be seen from Table 4, 30.998 L of water was treated at a flow rate of 23 ml/min while 15.644 L was treated at a flow rate of 40 ml/min and the breakthrough time also reduced from 2.25 to 6.5 h for 23 and 40 ml/min flow rates, respectively. This was due to the decrease in contact time between the fluoride and adsorbent at higher flow rate. As the adsorption rate was controlled by intraparticle diffusion, an early breakthrough occurs leading to a low bed adsorption capacity [26]. When the flow rate decreased, the contact time in the column was longer, and intra-particle diffusion then became effective. Thus, the adsorbate would have more time to diffuse into the particles of the adsorbent and a better adsorption capacity was obtained. The slope of the BDST plot would be higher for smaller flow rates.

Effect of EBCT

Fig. 7 as a plot of the adsorbent exhaustion rate against EBRT at various adsorbent bed height such as 20, 40, 60, and 100 cm. It could be seen from Fig. 7 that adsorbent exhaustion rate decreased with increasing EBRT. As shown in Table 5, the data of variable bed depth (20, 40, 60, and 100 cm) at a different flow rate (12, 23, and 40 ml/min) in a column reactor for the removal of fluoride. The data in Table 5 showed that EBRT, V and T increased with increasing bed depth. It was clear that when the EBRT increase with a flow rate, the bed volume would have to be longer, thus allowing more solution to be treated but resulting in a lower adsorbent exhaustion rate.

Thomas model

The data collected from continuous flow mode studies was used to determine the kinetic parameters using the Thomas model which was widely used for column studies. The Thomas model was used in describing the adsorption kinetics and evaluating the maximum solid phase concentration (q), and the rate constant (k). The linearized Thomas model plot was shown in Figs. 8-11. From the plot, the rate constant, k and the maximum solid phase concentration of the solute q were collected, and Table 6 shows summary of the linearized Thomas model parameters.

Characterization of java plum (S. cumini)

Scanning electron micrograph (SEM)

The surface morphology of the java plum (S. cumini) examined by SEM Fig. 12a and b that this adsorbent had irregular and porous surface. Difference in their surface porosity. Thus, the adsorbate would have more time to diffuse into the particles of the adsorbent and a better adsorption capacity was obtained. The slope of the BDST plot would be higher for smaller flow rates.

Energy dispersive x-ray analysis (EDAX)

EDAX of java plum (S. cumini) before and after adsorption of fluoride ions were shown in Fig. 13a and b. From Figs. 13a and b, it was clear

Table 5: Constant of BDST curve

| Q flow rate (ml/min cm²) | V (cm/h) | Slope | Intercept | Depth D (cm) | N (mg/cm³) | K (L/mg h) ×10⁻³ | X (mg/g) |
|-------------------------|---------|-------|-----------|-------------|------------|-----------------|---------|
| 0.1886                  | 11.316  | 0.464 | -2.29     | 4.963       | 0.105012   | 54.8479         | 0.358   |
| 0.3615                  | 21.69   | 0.266 | -3.771    | 14.071      | 0.11625    | 33.307          | 0.362   |
| 0.6287                  | 37.722  | 0.072 | -0.248    | 3.445       | 0.0543     | 506.45          | 0.1929  |

BDST: Bed depth service time

Table 6: Linearized Thomas model parameter at 7.5% breakthrough

| D (cm), C (mg/L), Q (ml/min) | K (L/mg min)×10⁻³ | Q (mg/g) | R² | Equation of line |
|-----------------------------|--------------------|----------|----|-----------------|
| 20, 20, 12                  | 0.257              | 0.596    | 0.884 | Y=−0.428x+3.83 |
| 40, 20, 12                  | 0.088              | 0.999    | 0.968 | Y=−0.148x+4.399 |
| 60, 20, 12                  | 0.120              | 2.476    | 0.874 | Y=−0.170x+6.314 |
| 100, 20, 12                 | 0.083              | 3.116    | 0.917 | Y=−0.138x+6.44 |
| 20, 20, 23                  | 0.124              | 1.654    | 0.888 | Y=−0.108x+2.68 |
| 40, 20, 23                  | 0.117              | 1.214    | 0.918 | Y=−0.102x+3.715 |
| 60, 20, 23                  | 0.136              | 0.893    | 0.990 | Y=−0.118x+4.74 |
| 100, 20, 23                 | 0.156              | 0.678    | 0.993 | Y=−0.136x+6.92 |
| 20, 20, 40                  | 0.81               | 0.483    | 0.993 | Y=−0.405x+2.936 |
| 40, 20, 40                  | 0.222              | 1.33     | 0.956 | Y=−0.222x+4.431 |
| 60, 20, 40                  | 0.312              | 0.6013   | 0.983 | Y=−0.156x+4.22 |
| 100, 20, 40                 | 0.282              | 0.0135   | 0.968 | Y=−0.141x+4.65 |

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that various elements such as carbon, oxygen and very small amount of calcium were present in virgin adsorbent but fluoride was not present there. When the EDAX of the adsorbent was carried out after the adsorption of fluorides ion, fluoride was present on the surface of adsorbent about 0.62 wt.% which confirmed the adsorption of fluoride by this adsorbent.

**Fourier transform infrared (FTIR)**

Functional groups present in biosorbent before and after adsorption was determined using FTIR spectroscopy (Thermo Nicolet, Magna 7600). The samples were prepared by pellet (pressed disk) method by mixing same amount of KBr in each sample. The selected spectral range was from 4000 to 400 (1/cm). Functional groups present on the surface of the peels were determined by the FTIR spectroscopy method.

Fig. 14a and b showed FTIR spectra of biosorbent, on the surface of adsorbent many functional groups were present (Table 7).

The range of different wave number assign the functional groups present in the adsorbent. The amine bond stretching lie in the wave range of 3400-3500, similarly for the very broad OH (2500-3300), NH$_2$ Scissoring stretching (1550-1650), alkanes (1370-1390), alcohols, and phenols ion stretching (970-1250).

**CONCLUSION**

In this study, we investigated the possibility of using java plum (S. cumini) seed for adsorption of fluoride in a packed bed column. The experimental results were encouraging and indicate that java plum (S. cumini) seed was a feasible option to use as a bio-adsorbent to remove fluoride in a fixed bed adsorption process.

The BDST model was successfully applied to analyze the column performance and to evaluate the model parameter. The BDST equations of linear relationship between the bed depth and the service time were obtained with $R^2=0.961$, 0.984 and 0.919 for 40, 23 and 40 ml/min flow rate, respectively.

The EBRT model which optimizes the EBRT and the sorbent utilization rate was successfully applied with optimum contact time greater than about 159.04, 276.59 and 530.14 min for 40, 23 and 12 ml/min flow rates,
respectively, with the corresponding usage rate of 154.72, 78.09 and 83.32 g/L. The optimum dose for batch system was 3.9 g/50 ml.

The application of Thomas model has shown that the adsorption capacity was strongly dependent on the flow rate, initial fluoride concentration, and bed depth and was greater under conditions of a lower concentration of fluoride, lower flow rate, and higher bed depth. Moreover, the Thomas rate constant decrease with increasing bed depth, decreasing initial concentration, and flow rate.

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Table 7: FTIR analysis for java plum (S. cumini) adsorbent in tabular form

| Wave number (1/cm) | 3400-3500 | 2500-3300 | 1550-1650 | 1370-1390 | 970-1250 |
|-------------------|-----------|-----------|-----------|-----------|----------|
| Compound/Groups   | Amines    | Carboxylic acids and derivatives | Amines | Alkanes | Alcohols and phenols |
| O-H (very Broad)  | NH₂ Scissoring (1° amines) | CH₂ and CH₃ deformation | C-O | | |

FTIR: Fourier transform infrared

Element | Weight % | Atomic % | Net Int | Error %
---|---|---|---|---|
C K | 48.97 | 56.83 | 65.9 | 7.16 |
O K | 48.33 | 42.11 | 51.49 | 11.41 |
Al K | 0.61 | 0.31 | 2.4 | 65.56 |
K K | 2.09 | 0.74 | 4.99 | 31.42 |

Element | Weight % | Atomic % | Net Int | Error %
---|---|---|---|---|
C K | 49.57 | 57.43 | 60.71 | 7.57 |
O K | 47.02 | 40.89 | 47.97 | 11.53 |
F K | 0.62 | 0.45 | 0.54 | 86.13 |
Mg K | 0.23 | 0.13 | 0.78 | 76.84 |
Al K | 0.63 | 0.32 | 2.38 | 65.93 |
Si K | 0.69 | 0.34 | 2.77 | 63.75 |
Ca K | 1.24 | 0.43 | 2.34 | 63.37 |

Fig. 13: (a) Energy dispersive X-ray analysis (EDAX) image of java plum (Syzygium cumini) seeds before biosorption, (b) EDAX image of java plum (S. cumini) seeds after biosorption

Fig. 14: (a) Fourier transform infrared (FTIR) analysis of java plum (Syzygium cumini) before adsorption of fluoride, (b) FTIR analysis of java plum (S. cumini) after adsorption of fluoride
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