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Research Article

Fabrication of an adsorbent from *Ficus racemosa* leaf biomass for fluoride removal and its preparation cost analysis

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Abstract

This study aims to prepare an adsorptive agent from the cluster fig (*Ficus racemosa*) leaf biomass and its utilization for the elimination of fluoride ions from the synthetic water. A carbon material developed from Leaf biomass was impregnated with zirconium at temperature (300 ºC). Scanning electron microscopy (SEM) and Fourier transformed infrared spectroscopy (FTIR) were used to analyze the appearance of surface structure and present functional groups on its surface. Determination of zero point charge (pHzpc) of the adsorbent particles also was conducted by salt addition methods. Effect of adsorbent amount, pH of the solution, interaction time, and starting fluoride ion concentration on adsorption percentage were studied at room temperature. Under optimized conditions including 4 g/L adsorbent dose, 10 mg/L initial concentration of fluoride, and neutral pH, the maximum adsorption percentage of fluoride was found to be 84.5%. The maximum adsorption capacity of the adsorbent was obtained 2.12 mg/g. The adsorbent having good adsorption performance can be applied for the defluoridation of water. The preparation cost of the prepared adsorbent also was estimated in this study.

Keywords: *Ficus racemosa*; adsorption; fluoride; fluorosis

1. Introduction

Fluorine is present in different geological conditions and 0.08% fluorine content is present as minerals form in the earth's crust (Bhatnagar et al., 2011). The minerals such as fluorapatite, fluorite, cryolite, and sellaite are the primary and rich sources of fluoride that come into the water system after the dissolution of these minerals (Fuoco et al., 2021; Pant et al., 2021). Glass industry, electroplating plant, photovoltaic industry, metal smelting, and coke production units are the anthropogenic sources of fluoride that contaminate the water (Gai and Deng 2021). Overall, about 20 nations of the world like the Rift Valley of Africa, USA, Asia including Srilanka, India, China and Pakistan, where fluorosis became endemic. The groundwater of the 17 states of India such as Tamil Nadu, Andhra Pradesh, Rajasthan, Gujarat, and Uttar Pradesh are badly affected by fluoride, and people of these areas are at high risk of fluorosis (Choubisa et al, 2018). More than one million population in India are facing skeletal fluorosis endemically. Fluoride concentration should not become more than 1.5 mg/L in drinking water and this concentration has been set by the World Health Organization (WHO). While, 15 mg/L is the permissible limit for the average national effluent standard (Lacson et al., 2021). About 80% of villages of Asia and Africa facing endemic fluorosis and approximately 100 million people of these villages take fluoride containing water having a high concentration of more than 1.5 mg/L (Srivastava and Flora, 2020). Therefore, presently removal of fluoride from the water became a need for human health using feasible and low-cost technologies. Thus, various fluoride removal technologies came with advancement in past years. These are nanofiltration, ultrafiltration, reverse osmosis, electrodialysis, and electrocoagulation with high efficiency (Singh et al., 2016; Fadaei et al., 2021). But these are technically complex, have a high cost, and are not easily affordable by a lot of rural and poor communities. Due to wide-scale application and their easy operation adsorption-based removal is known more popular method. Adsorbents like activated carbon, biochar, which are originated from the low-cost materials (crop residue, food waste, municipality waste, and forest residue) are very attractive their use as raw material and this biomass are easily available in the local areas. In this work, *Ficus racemosa* leaf biomass is a woodland residue that was used for the preparation of modified carbon as adsorbent.

2. Materials and methods

2.1 Materials

Ficus racemosa leaf biomass was collected from woodland near Faridpur village, Ambedkar Nagar, Uttar Pradesh, India. Analytical grade chemicals such as hydrochloric acid (HCl) sodium hydroxide (NaOH), Zirconium oxychloride, Ion strengthen adjuster, and stock solution of fluoride (1000 mg/L) were got from fisher scientific, India.

2.2 Adsorbent preparation

Dried leaves of *Ficus racemosa* were ground in a mixture grinder and sieved by using a sieve having 250 micrometer pore size to obtained uniform fine powder. The leaf powder was subjected to pyrolyzed at 500 ºC in a furnace for 1 h. A 40 g prepared carbon was stirred with 100 mL solution of 0.1M zirconium oxychloride. The solid content was separated from the solution and calcined at 300 ºC in a muffle furnace for 60 min (**Figure 1**). The heated solid was cleaned with distilled water many times and it was subjected to drying in the oven overnight. The prepared zirconium modified *Ficus racemosa* based material was denoted as FR-ZrAC and it was stored in a bottle for adsorption purposes.

2.3 Characterization of FR-ZrAC

Microstructure appearance and elemental constituents of the FR-ZrAC before and after fluoride adsorption was investigated by using Scanning Electron microscopy armed with energy-dispersive x-ray spectroscopy (EDS) (Model: JSM 4490 JEOL, JAPAN). Confirmation of the functional groups on FR-ZrAC was performed by FTIR (Model: Nicolet 6700, Thermo-scientific, USA) under the spectral range 400 to 4000 cm^{-1}. Determination of the pHzpc of FR-Zr-AC was done by the salt addition method (Yadav and Jagadevan, 2021).

Fig. 1 Preparation of *Ficus racemosa* leaves based adsorbent

2.4 Fluoride adsorption experiment

The fluoride adsorption study was performed with an aqueous solution of fluoride by using FR-ZrAC. All batch adsorption experiments were conducted for optimization of conditions like equilibrium time, pH of the solution and temperature. A decided dose was kept with 50 mL solution in a 250 mL Erlenmeyer flask and shaken in an orbital shaker at 80 rpm. After the desired time, the sample was collected and filtered by using the Whatman filter. A 0.5 mL Ion strengthen adjuster (ISA) solution was put in the filtered sample. Then the remaining concentration of fluoride ion in the solution was confirmed by a selective ion meter (Eutech ION 6^+ THERMO SCIENTIFIC, USA). The fluoride removal percent and adsorption capacity of FR-ZrAC were determined by the following equations (1) and (2), respectively, as reported in previous study (Bhan et al., 2021).

$$R\ (\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \qquad (1)$$

$$Adsorption\ Capacity = \frac{(C_0 - C_t)}{m} V \qquad (2)$$

Where C_0 and C_t are starting concentration and the final concentration of fluoride at any time, respectively. V and m are the volume of solution and dried mass of FR-ZrAC, respectively.

3. Results and discussion

3.1 Characterization of synthesized adsorbent

Activated carbon appeared rough and irregular in shape under SEM **(Fig. 2).** After the adsorption of fluoride, it is seen as bigger due to the clumping of particles. The peak of fluoride on the EDS spectrum revealed that fluoride ions adsorbed on the surface of activated carbon developed from *Ficus racemosa*. FTIR spectrum as shown in **Fig. 3b**, revealed that vibration of Zr-OH depicted at wavenumbers 651.5 cm^{-1} (Das and De, 2015). The peak at 1227.8 cm^{-1} and 1699 cm^{-1} represents the vibration of the C-OH bond (Alzaydien et a., 2016) and C=O bond (Sharma and Naushad, 2020), respectively. The peaks 1581.4 cm^{-1} are represented to the bond of C=C in FR-ZrAC and a small peak of Zr-F was appeared at 1402.1 cm^{-1} after fluoride adsorption (Mullick and Neogi, 2018). Wavenumbers 3400 cm^{-1} represent -OH bond while the intensity of this peak was found altered after fluoride adsorption. The pHzpc of the FR-ZrAC was observed to be 5.8 (**Fig. 3a**), at this point the adsorbent surface has electrically neutral charge. The pHzpc determines that the adsorbent surface is acidic or basic and this property of the adsorbent allows for electrostatic interaction between adsorbent and adsorbate. The surface of FR-ZrAC was acidic in nature that was suitable for adsorption of fluoride.

3.2 Adsorption experiments

3.2.1 Effect of FR-ZrAC amount

The amount of FR-ZrAC for fluoride adsorption was performed at varied doses from 0.5 to 4.5 g/L under the optimum conditions (fluoride concentration-10 mg/L, pH of the solution-7, and temperature-25 ºC) as shown in **Figure 4a**. The results observed that with the increasing an amount of FR-ZrAC, the removal percentage of fluoride was increased while adsorption capacity of its decreased. Fluoride adsorption was found 32%, 73%, 84.5% with the doses 0.5 g/L, 2.5 g/L and 4 g/L, respectively. Removal (in percentage) of Fluoride got equilibrium at 4 g/L after this dose, there was no significant adsorption of its happened. Therefore, 4 g/L dose was selected for the further studies including contact time, fluoride ion solution, and pH.

The removal percentage was achieved 92.1%, 84%, 67.7% and 51% for initial fluoride concentration of 5 ppm, 10 ppm, 15 ppm and 20 ppm, respectively. At low concentration, more interaction of fluoride ions on the adsorptive sites and it was decreased at higher concentrations because of active sites saturated with fluoride ions.

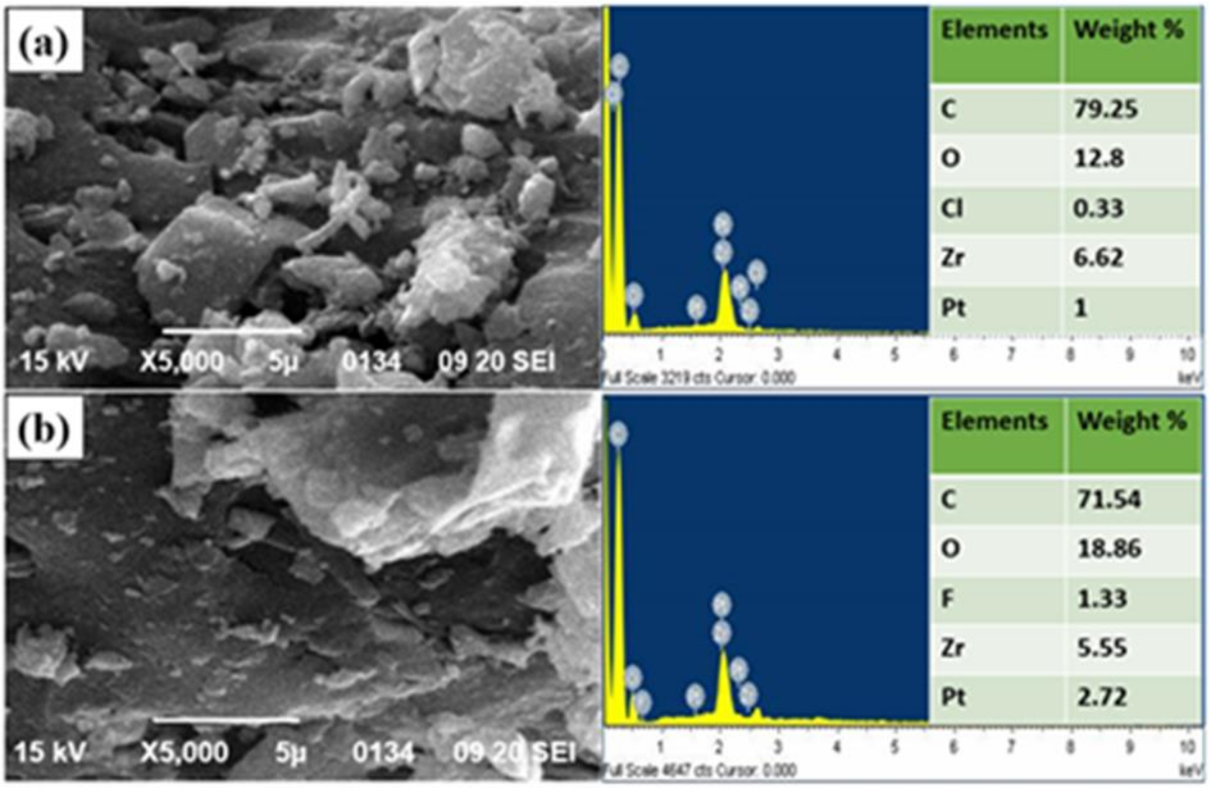

Fig. 2 SEM and EDS analysis of FR-ZrAC (a) before and (b) after fluoride adsorption

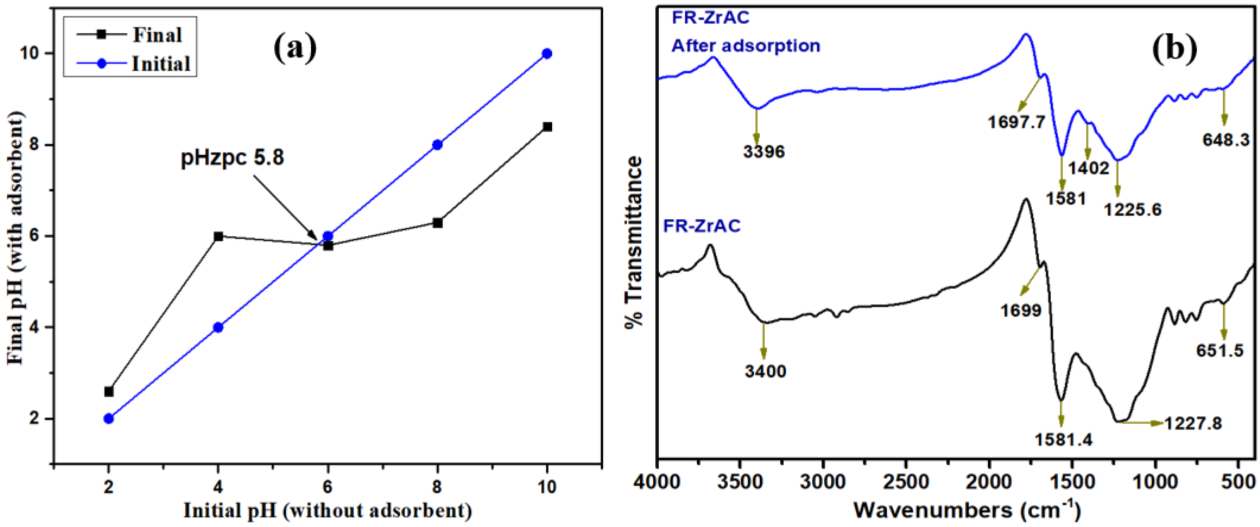

Fig. 3 Point zero charge (a) and FTIR spectrum of adsorbent (b).

3.2.3 Effect of pH of the solution

It can be observed from **Figure 4c** that fluoride removal is strongly influenced by pH. Adsorption of fluoride increase with a lowering of the pH of the solution. At pH 7 removal percentage was found to be 84.5% while it was 90.5% and 47.7% at pH 4 and 12, respectively. In the acidic condition, the adsorbent surface gets protonated by H^+ ions and the positively charged surface attract more fluoride ions while basic condition attributed to fluoride ion repulsion that decreases adsorption. The overall cost of 1 kg prepared adsorbent was evaluated to be 581.72 INR. If the material was dried under the sun and air then the total cost could be 462.2 INR, this cost is lower than commercially available adsorbent having cost 600 INR (Mohanta and Ahmaruzzaman 2018).

4. Preparation cost estimation of 1 kg adsorbent

Evaluation of cost-effectiveness and feasibility is a very essential step for adsorbent preparation and water treatment for the rural community. The cost of adsorbent synthesis depends on the consumed amount of chemicals, electric power (kWh), and overhead cost. The details of these expenditures are mentioned in **Table 1**.

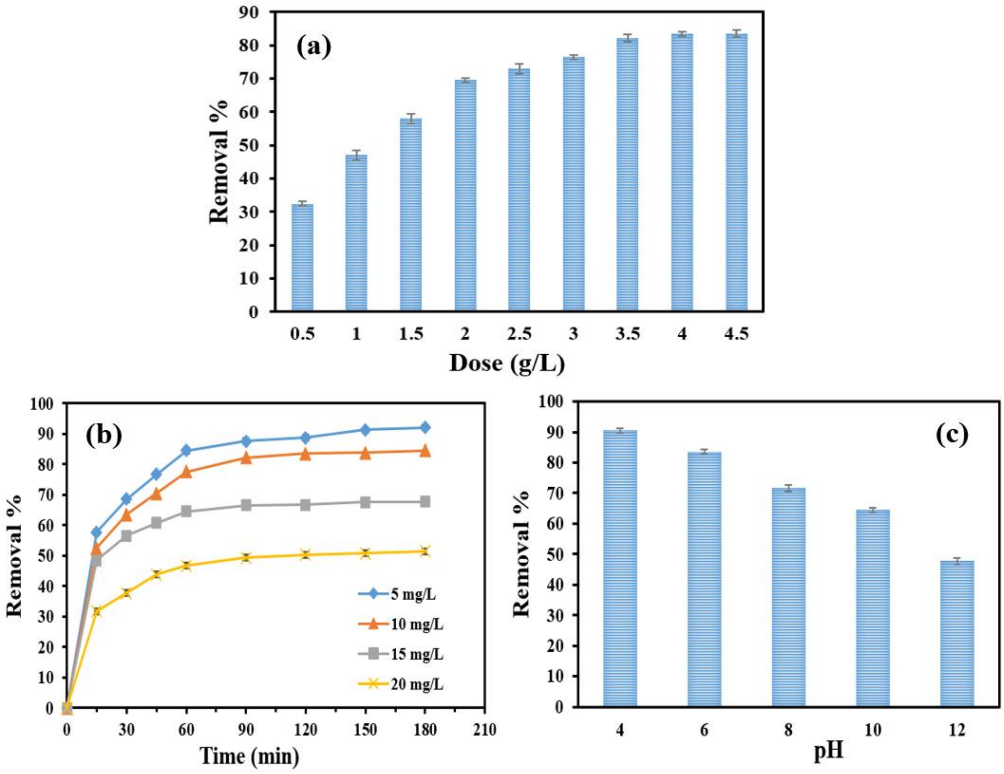

Fig. 4 Effect of FR-ZrAC dose (a), contact time and initial concentration of fluoride (b), and pH of the solution.

Table 1: Preparation cost of 1 kg adsorbent from *Ficus racemosa* leaves biomass

S.N.	Chemicals/Process	Amount used	Unit cost (INR)	Net cost (INR)
1.	Leaves biomass	0	0	0
2.	Mixture grinder	1.60 kWh (2 h)	6/kWh	9.6
3.	Magnetic stir	0.3 kWh (400 rpm and 1 h)	6/kWh	1.8
4.	Oven-dried	19.92 kWh (105 ºC for 24 h)	6/kWh	119.52
	Sun + air dried	0.00	-	0.00
5.	Muffle furnace	8 kWh (500 ºC and 300 ºC; 2 h)	6/kWh	48
6.	Zirconium oxychloride ($ZrOCl_2.8H_2O$)	72 g	4.86/g	349.92
7.	Net cost			528.84
8.	Overhead charge (10% of the net cost)			52.88
Total cost				**581.72**

5. Conclusion

This study reported the preparation of activated carbon from *Ficus racemosa* leaf biomass with zirconium impregnation at 300ºC temperature. The cost analysis was also included in this study. EDS analysis represents the binding of zirconium on the surface of FR-ZrAC and it also successfully adsorbed fluoride ions. It was perceived that pH of the solution strongly affects the adsorption capacity of fluoride due to the alteration in electrostatic force between the net surface charge of the adsorbent and electronegative fluoride ions. The maximum adsorption percentage was attained 90.5% at pH 4, while the minimum adsorption percentage was found 47.7% at pH 12. About 84% of the fluoride was found to be adsorbed on FR-ZrAC surface in 120 min at 25 ºC and neutral pH. The preparation cost of 1 kg adsorbent was estimated based on waste biomass, the chemical used, electricity, and other overhead costs. The cost of 1 kg adsorbent was evaluated INR 581 which is lower than commercially available adsorbent. A large amount of woodland leaf biomass may be applied as stock material for the preparation of adsorbents.

Author contributions

Chandra Bhan: Methodology, Investigation, Writing - original draft. Jiwan Singh: Supervision, review & editing.

Conflicts of interest

The authors declare that they have no known conflict of interest.

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