

Exploring Low-Cost Adsorbents for Efficient Fluoride Removal

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Abstract

Fluoride (F) contamination in water poses a significant health risk, emphasizing the need for effective removal methods. This study examines the efficacy of low-cost adsorbents: Rice Husk (RH), Broken bricks (BB), and Activated Carbon (AC)—for F removal. Batch experiments were conducted to characterize these adsorbents and evaluate their performance across varying pH levels. Results reveal a substantial impact of pH on F removal efficiency, with RH, BB, and AC exhibiting optimal rates at pH 4, 2, and 6, respectively. This underscores the influence of pH-dependent surface charge characteristics and F ion speciation on removal efficiencies. The results of competition studies revealed that the adsorbent BB demonstrates the highest affinity for F ions compared to RH and AC suggesting that BB is more effective at adsorbing F ions compared to the other adsorbents. Furthermore, analysis of adsorption kinetics and optimal dosages uncovers differences in adsorption mechanisms among the adsorbents. RH adheres to the Langmuir model, while BB follows the Freundlich model, and AC outperforms according to both models. These findings underscore variations in adsorption capacities, affinities, and mechanisms, with AC emerging as a promising option for efficient F removal. Nonetheless, further research is necessary to fully comprehend the nuanced mechanisms behind pH-dependent F removal and to explore potential pre-treatment strategies to enhance RH and BB's adsorption capacity. In summary, this study provides valuable insights into developing efficient F removal strategies using economical adsorbents.

Keywords: F, Water, Water quality, Low-cost adsorbent,

1. Introduction

F contamination in water sources poses a pressing public health concern worldwide. Excessive F intake has been linked to various health issues, such as dental and skeletal fluorosis, which can have severe consequences [1-2], especially in regions with limited access to clean water. For instance, in regions like India, China, Bangladesh and parts of Africa, high levels of naturally occurring F in groundwater have led to widespread health problems among populations relying on these sources for drinking water [3-4].

Prolonged ingestion of excessive F can also lead to chronic kidney effects [5]. The World Health Organization (WHO) recommends a F level of 0.7 parts per million (ppm) from infancy to 16 years of age. Additionally, to prevent dental issues, the WHO suggests a maximum acceptable concentration of F ions in drinking water to be 1.5 ppm [6]. Given Sri Lanka's location in a tropical humid zone, the per capita water consumption is relatively high. For instance, 50% of the dry zone areas in Sri Lanka have F levels above 1 ppm [7]. Consequently, the desired F level in groundwater in Sri Lanka is set at 1.0 mg/L as per the Sri Lankan Specification for Potable Water SLS 614-2013. Hence, it is imperative to eliminate excess F from drinking water.

In addressing this issue, various methods have been employed for F removal during water treatment processes [9]. These methods include coagulation,

precipitation, membrane filtration, and ion exchange [10]. However, these techniques often require sophisticated infrastructure and can be costly to implement, particularly in resource-constrained settings. Furthermore, absorption well proven highly studied area briefly justify why this study was performed over other methods.

Adsorption, as a water treatment method, has gained attention due to its effectiveness in removing F from water at reasonably low cost. Adsorption involves the attachment of F ions to the surface of a solid material, known as an adsorbent, thereby reducing their concentration in the water. Among the various adsorbents investigated for F removal, low-cost materials have garnered significant interest due to their affordability and accessibility.

The efficiency of various adsorbents in removing F from water has been extensively examined in prior studies [11-16]. However, a notable observation is that most of these studies have not thoroughly investigated the competitive effects of other ions present in water during the adsorption process. For instance, Vardhan and Karthikeyan (2011) explored the efficiency of rice husk (RH) in removing F and identified chloride, nitrate, magnesium, carbonate, sodium, and sulphate as competitive anions and cations [11]. Similarly, Memon et al (2021) evaluated the efficacy of biochar (BB) and found it to be 90.3%, yet they did not delve into the influence of competitive ions [12]. Bandewar et al. (2015) conducted a study focusing on the efficacy of activated carbon as an adsorbent for F removal. Similarly, Koteswara and Mallikarjun (2014) reported

the potential of various low-cost adsorbents for this purpose [13]. However, despite their findings on the effectiveness of these adsorbents, neither study explored the competitive effects of other ions present in water during the adsorption process. Additionally, Ali, Ahmad, and Singh (2016) examined bottom ash and sugar cane bagasse as potential adsorbents, however, this study did not explore ion competition, revealing a gap in the current research concerning the comprehensive understanding of F removal mechanisms when competing ions are present [12].

In the majority of the aforementioned studies that utilized low-cost adsorbents, comprehensive isotherm studies are lacking. Therefore, despite the effectiveness of these adsorbents in F removal, the absence of comprehensive studies on ion competition and isotherms limit our understanding of the factors affecting F adsorption. Therefore, further research is essential to clarify the complex dynamics of ion competition, conduct detailed isotherm studies, and understand their impact on F removal efficiency with low cost adsorbents. This will contribute to the advancement of more effective water treatment strategies.

In this study, we address this gap by focusing on the effect of common ion competition on F removal efficiency and the isotherms using three specific adsorbents. In this study, we focused on evaluating the efficacy of three low-cost adsorbents: Rice Husk (RH), Broken Bricks (BB), and Activated Carbon (AC) for F removal from water. The selection of these adsorbents stems from their abundance and cost-effectiveness, making them promising candidates for addressing F contamination in resource-limited settings. By investigating the performance of these adsorbents under varying pH conditions and analysing their adsorption mechanisms, and completions with other common ions which present in water, this study aims to provide valuable insights into the development of cost-effective and efficient F removal strategies using readily available materials.

2. Materials and Methods

2.1 Preparation of local available adsorbent materials

RH, BB, and AC were selected as the locally available low-cost adsorbents. Locally sourced rice husks were gathered from Mahiyaganaya, Sri Lanka, and processed as follows. Prior to use, each raw adsorbent was sized appropriately, washed with distilled water, and then dried. Broken bricks were obtained from discarded brick fragments, crushed, and collected as brick powder passing through a 300 μm sieve. The collected powder was washed with distilled water until the effluent became clear, followed by drying in an oven at 105°C for 12 hours [17]. Activated carbon was procured from the local market, sieved through a 150 μm sieve, and the particles that passed through were collected [17].

2.2 Stock solution and glassware preparation

A stock solution containing 100 mg/l of F was created by dissolving 221 mg of Sodium F (NaF) in 1000 ml of distilled water. F solutions of desired concentrations were then prepared using this stock solution [11]. Glassware used in the experiment was soaked overnight in a F solution of 5.0 mg/L to reduce F absorption. Subsequently, the glassware was rinsed with nitric acid and distilled water before use.

2.3 Batch experiments

Batch sorption experiments were conducted to investigate the influence of various controlling parameters, including pH value, contact time, sorbent dosage, and competition from other anions.

a) Effect of pH

A test solution containing 6.5 mg/L of fluoride (F) was prepared by diluting the appropriate amount of a 100 mg/L fluoride stock solution with distilled water. This concentration aligns with the higher levels of fluoride often found in groundwater-affected areas of Sri Lanka, where concentrations can exceed 5 mg/L. [18]. From this 6.5 mg/L F simulated solution, 50 mg/L solutions were prepared and transferred into prepared glassware. The impact of pH on F adsorption was examined by adjusting the pH of the test solutions for each adsorbent, utilizing 0.1M H_2SO_4 and 0.1M NaOH, across a pH range of 2 to 12. Each sample was equilibrated by shaking in a horizontal shaker at 200 rpm for a contact time of 1 hour. Following the shaking period, the residual F concentration was measured using colorimetric method.

b) Effect adsorbent dosage

The impact of adsorbent dosage was examined by conducting the same procedure while varying the adsorbent dosage, maintaining optimum pH values for each adsorbent, and utilizing a contact time of 1 hour. The adsorbent dosages were adjusted within the ranges of 2 to 18 g/L for RB, 10 to 50 g/L for BB, and 6 to 30 g/L for AC. The pH was maintained at the previously determined optimum value, and the contact time was kept consistent at 1 hour. After 1 hr contact period, the suspensions were filtered through filter papers, and the remaining F concentration was measured using a colorimetric method.

c) Contact time determination

To determine the optimal contact time, F adsorption by the adsorbent was studied under the optimum pH and dosage conditions. The equilibrium of adsorption was established when the change in F removal efficiency was less than 5%.

d) Competition studies for naturally available ions

The impact of naturally available ions competitive ions on F removal was examined by varying ion concentrations from 50 mg/L to 300 mg/L, while maintaining optimal pH values, contact times, and adsorbent dosages for each adsorbent. The adsorption kinetics of RH, BB, and AC were explored concerning chloride (Cl⁻), nitrate (NO₃⁻), sulfate (SO₄²⁻), and phosphate (PO₄³⁻). The distribution coefficient K_d is calculated using the equation 1 [20-21]:

$$K_d = \frac{q_e \text{ mmol/g}}{C_e \text{ mmol/mL}} \quad (1)$$

Where; K_d , q_e , and C_e are distribution coefficients, amount of F adsorbed, and equilibrium concentration, respectively.

2.4. Adsorption isotherms

In the adsorption isotherm analysis, the Langmuir and Freundlich isotherms were studied under different adsorbent and adsorbate concentrations. The required parameters were calculated by plotting the Langmuir and Freundlich isotherm curves as described below as previously reported by Jamode, sapkal, and Jamode (2013) and Ayawei, Ebelegi, and Wankaso (2017), respectively [22-23].

a) Langmuir Isotherm

$$\left(\frac{1}{q_e}\right) = \left(\frac{1}{Q_0}\right) + \left(\frac{1}{bQ_0}\right)\left(\frac{1}{C_e}\right) \quad (2)$$

Where, q_e , and C_e are amount of F adsorbed, and equilibrium concentration, respectively and Q_0 and b are the Langmuir constants related to adsorption capacity and energy, respectively.

b) Freundlich Isotherm

$$\log(q_e) = \log(K_f) + \left(\frac{1}{n}\right)\log(C_e) \quad (3)$$

Where, K_f and n are constants in Freundlich isotherm and q_e , and C_e are distribution coefficients amount of F adsorbed, and equilibrium concentration, respectively.

3. Results and Discussions.

3.1 Effect of pH

The impact of varying pH levels on F removal using three distinct adsorbents (RH, BB, and AC) is illustrated in Fig. 1. As depicted in Fig. 1 demonstrates that the highest efficiencies in F removal were achieved at pH

levels of 4, 2, and 6 for RH, BB, and AC, respectively. These findings suggest that pH significantly influences the effectiveness of the adsorbents in removing F from the solution. The observed variations in removal efficiencies at different pH levels could be attributed to the pH-dependent surface charge characteristics of the adsorbents, which affect their affinity for F ions.

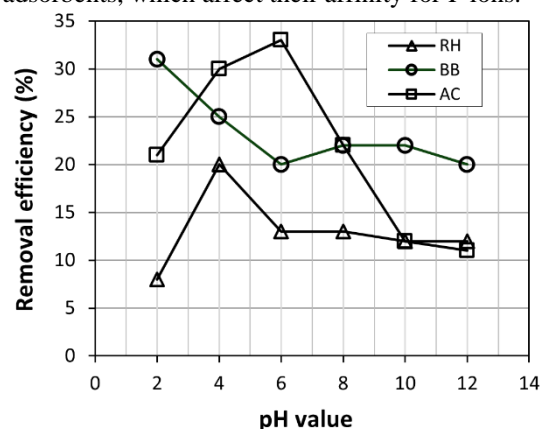


Fig. 1. Effect of initial pH value on removal efficiency (contact time was 120 mins, dosages of for RH, BB, and AC are 15, 24, and 15 g/L, respectively and initial F concentration was 6.5 mg/L).

In solutions with a pH below the zero point charge (ZPC) of a surface, the surface tends to exhibit a positive charge owing to the adsorption of excess H⁺ ions [24]. This creates a favorable environment for the attachment of negatively charged species onto the surface. Consequently, under acidic conditions, there is an increased propensity for the removal of F ions. However, it's noteworthy that when the pH value becomes extremely low, F ions may undergo conversion into neutral hydrogen F. This transformation can weaken the ion exchange process, potentially affecting the efficiency of F removal. Thus, while acidic conditions generally promote the adsorption of F ions onto surfaces, excessively low pH levels may introduce complexities in the removal mechanisms, necessitating careful consideration of pH control in F remediation strategies.

3.2 Contact time

The duration of contact time necessary for F removal was investigated through continuous monitoring of F concentration over time. As depicted in Fig. 2, the optimal contact times for achieving maximum F removal for RH, BB, and AC were found to be 150 minutes, 120 minutes, and 120 minutes, respectively. These results indicate that different adsorbents require varying durations of contact time to remove F from the solution effectively. The variations in contact time requirements could be attributed to differences in the surface properties, pore structures, and chemical compositions of the adsorbents, which influence their adsorption kinetics and capacity for F ions. Further investigation into the specific mechanisms governing the adsorption process is warranted to understand better the observed differences in contact time among the adsorbents.

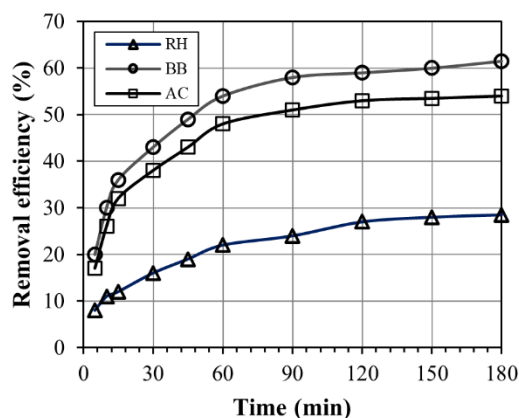


Fig. 2. The variation of F- removal efficiency of different adsorbents with time (dosages of for RH, BB, and AC are 15, 24, and 15 g/L, respectively and initial F concentration was 6.5 mg/L).

3.3 Adsorbent dosage

The study examined the efficiency of F removal at various dosages of different adsorbents. As illustrated in Fig. 3., nearly maximum removal efficiencies for F were achieved at dosages corresponding to 15, 24, and 15 g/L for RH, BB, and AC, respectively. These findings suggest that the effectiveness of F removal is influenced by the dosage of the adsorbent utilized.

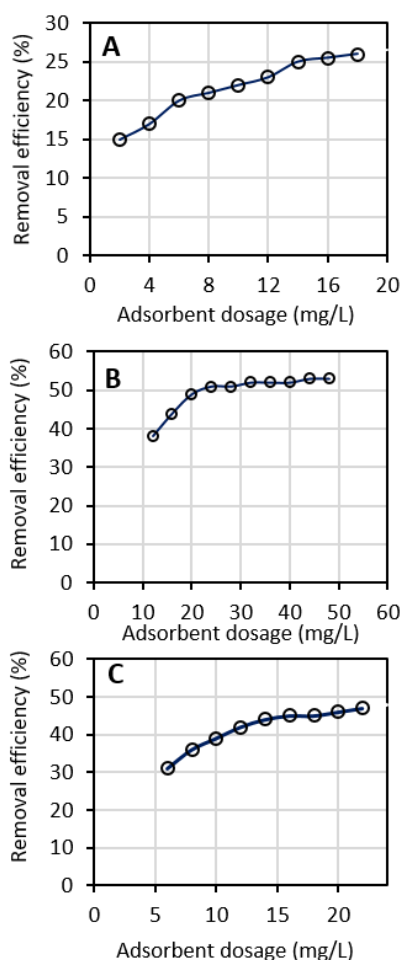


Fig. 3: Variation of F- removal efficiency with adsorbent dosages (A) RH, (B) BB, and (C) AC (initial F concentration is 6.5 mg/L).

Several studies have reported varying degrees of F removal efficiencies using different adsorbents. For

instance, Vardhan and Karthikeyan (2011) demonstrated an 83% removal efficiency [11], while Bandewar et al. (2015) achieved a removal efficiency of 72% using AC [13]. Additionally, Manjunath et al (2014) obtained a notably higher removal efficiency of 48 – 90 % using BB [25]. These findings highlight the diverse efficacy of various adsorbents in mitigating F contamination from aqueous solutions. The differences in removal efficiencies could be attributed to several factors, including the specific surface characteristics, pore structures, and functional groups of the adsorbents, as well as variations in experimental conditions such as pH, temperature, contact time, and initial F concentration. Overall, these studies underscore the importance of selecting appropriate adsorbents and optimizing experimental parameters to achieve efficient F removal from water sources.

Table 1 delineates the optimal conditions for F removal using three distinct adsorbents: RH, BB, and AC. RH exhibits its highest F removal efficiency at a pH of 4, requiring an adsorbent dosage of 15 mg/l and a contact time of 120 minutes. In contrast, BB achieves maximum F removal efficiency at a pH of 2, with an optimal adsorbent dosage of 24 mg/l and a contact time of 90 minutes. AC demonstrates its peak performance at a pH of 6, with an adsorbent dosage of 15 mg/l and a contact time of 120 minutes.

Table 1
Summary of optimal condition for F removal for different adsorbents.

Adsorbent	Optimum pH Value	Optimum Adsorbent Dosage(mg/l)	Optimum Contact Time (min)
RH	4	15	120
BB	2	24	90
AC	6	15	120

These findings highlight the variability in optimal pH, adsorbent dosage, and contact time among the three adsorbents, reflecting differences in their surface properties, pore structures, and chemical compositions. Such insights are crucial for designing efficient F removal strategies tailored to the characteristics of specific adsorbents in water treatment applications.

3.4 Competition with other ions

The study investigated the competitive adsorption of F ions with other common ions present in water using different adsorbents. Fig. 4 illustrates that the adsorption capacities of RH and AC remain relatively stable even in the presence of competing ions. However, the F adsorption capacity of BB appears to be influenced by certain ions commonly found in water. To gain a deeper understanding of the selectivity of these low-cost adsorbents, distribution coefficients (K_d) were calculated and are presented in Table 2. These K_d values provide insights into the relative affinity of the adsorbents for F ions compared to other competing ions. These results will offer valuable information on the selectivity and effectiveness of the chosen adsorbents in

removing F from water containing various competing ions, contributing to the development of more efficient water treatment strategies.

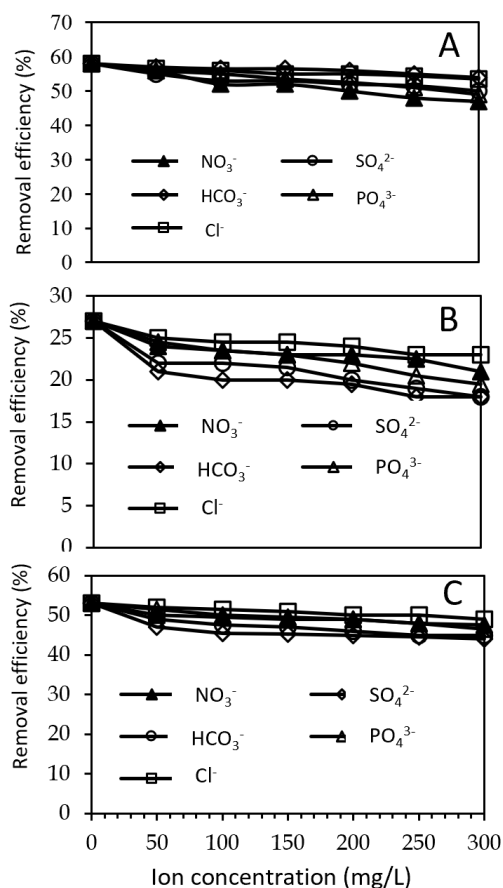


Fig. 4: The F removal efficiencies of adsorbent (A) BB, (B) RH, and (C) AC, with the presence of other common ions (initial F concentration is 6.5 mg/L).

These K_d values provide insights into the relative affinity of each adsorbent (RH, BB, and AC) for different ions compared to F ions. Higher K_d values indicate a greater affinity for the respective ion, while lower values suggest a lesser affinity. The data indicates that BB exhibits the highest affinity for F ions compared to RH and AC, as evidenced by the significantly higher K_d value for F with BB. Further, each adsorbent material's selectivity order of each anion is shown below.

For RH: $F^- > SO_4^{2-} > PO_4^{3-} > NO_3^- > Cl^-$

For BB: $F^- > NO_3^- > PO_4^{3-} > SO_4^{2-} > Cl^-$

For AC: $F^- > SO_4^{2-} > PO_4^{3-} > NO_3^- > Cl^-$

Vardhan and Karthikeyan (2011) previously reported a selectivity order for competing ions, indicating Carbonates > Nitrates > Sulphates > Chlorides, which bears resemblance to the findings of our current study [11]. This alignment suggests a consistent trend in the adsorption behaviour of these ions onto the RH surface. Such concurrence across studies underscores the robustness of the observed phenomena and lends credence to our own investigation.

Table 2

The K_d values for different adsorbent with the presence of various common ions.

Competing ion	Distribution coefficient (K_d)		
	RH	BB	AC
NO_3^-	5.06	8.65	6.98
SO_4^{2-}	7.32	6.67	10.71
PO_4^{3-}	6.21	7.35	8.1
Cl^-	3.46	4.03	5.23
F^-	24.66	57.54	75.18

3.1 Adsorption isotherms

As explained in Section 2.4, the adsorption isotherms were investigated for the tested low-cost adsorbents. Langmuir and Freundlich (Fig.5) isotherms were used to describe the equilibrium data, and results are shown in Fig. 5A and 5B. The Langmuir and Freundlich isotherm constants were determined by analysing the slopes and intercepts of their respective graphs. These isotherms exhibited a strong alignment with the experimental data, as evidenced by their higher distribution coefficients (K_d) are shown in Table 2.

The favourable nature of adsorptions is evident, with bond energies escalating alongside surface density for RH, AC, and BB. Analysing the adsorption isotherms reveals that BB adheres to the Freundlich model, RH adheres to the Langmuir model, and AC adheres to the Freundlich model.

As shown in Fig 5, the from Langmuir and Freundlich isotherms for three distinct materials: RH, AC, and BB. all materials exhibit high correlation coefficients (R^2) with the Langmuir model, implying a good fit between theoretical predictions and experimental data. Contrastingly, when considering the Freundlich model, AC emerges as the most favourable adsorbent. These findings suggest that AC holds promise as a highly efficient adsorbent, potentially outperforming RH and BB in various adsorption applications. Therefore, in order to enhance the adsorption capacity of RH and BB, an additional pre-treatment such as protonation is to be investigated in a future study.

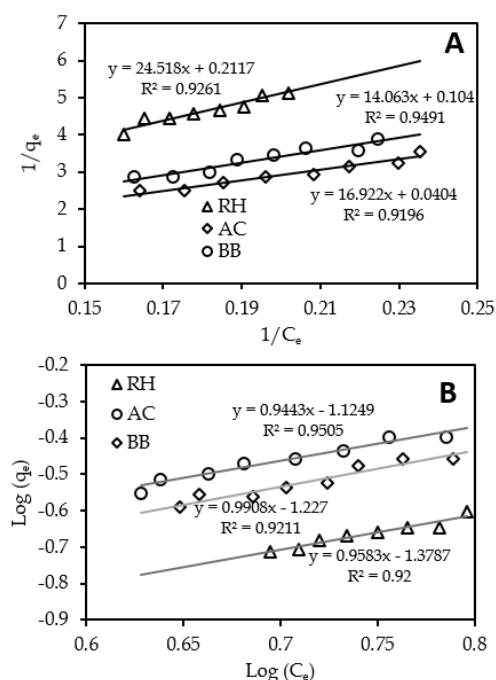


Fig. 5. The isotherms (A) Langmuir and (B) Freundlich for F adsorption by RH, BB and AC

3.5 Adsorption mechanisms

The adsorption mechanisms of BB, RH, and AAC can be discerned by examining the parameters obtained from Langmuir and Freundlich isotherms. From the Langmuir model, it's evident that BB exhibits the highest maximum adsorption capacity among the three materials, indicating its propensity to adsorb a significant amount of solute. AC, on the other hand, demonstrates the lowest Langmuir constant, suggesting a strong affinity for adsorption. Despite variations in their capacities and affinities, all materials show a good fit with the Langmuir model, indicating a multilayer adsorption mechanism on surfaces with a finite number of identical sites [26]. In contrast, the Freundlich model reveals that AC possesses the highest Freundlich constant, indicating superior adsorption capacity compared to RH and BB. Additionally, AC exhibits the highest Freundlich, signifying enhanced adsorption intensity and suggesting a heterogeneous or multilayer adsorption mechanism [27]. These findings suggest that AC may offer advantages over RH and BB in adsorption applications, owing to its higher capacity, stronger affinity, and enhanced intensity of adsorption

4. Conclusions

This study investigates the effectiveness of RH, BB, and AC as potential adsorbents for F removal. The pH level significantly impacts removal efficiency, with optimal rates observed at pH 4 for RH, pH 2 for BB, and pH 6 for AC. These results emphasize the critical role of pH in adsorption performance, particularly in relation to pH-dependent surface charge characteristics and fluoride ion speciation, which are key factors influencing removal efficiencies and the practical

application of these adsorbents. Competitive studies revealed that BB has a stronger affinity for fluoride ions compared to RH and AC, suggesting that BB is more effective in adsorbing fluoride ions than the other adsorbents. Additionally, a detailed analysis of adsorption kinetics and optimal dosages offers deeper insights into the mechanisms driving fluoride adsorption. While RH follows the Langmuir model and BB aligns with the Freundlich model, AC shows strong performance according to both models. These differences highlight variations in adsorption capacities, affinities, and mechanisms among the adsorbents, with AC emerging as a particularly promising candidate due to its robust adsorption capacity and high affinity.

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