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Reduction of Fluoride Ion from Water by Utilization of Natural Bioadsorbents: A Comparative Study

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Abstract:

The current study primarily focused on experimental investigation of fluoride ion removal from water using multiple low-cost adsorbents, followed by adsorption isotherms and thermal kinetic models. The experimentation was performed in batches at a constant residual concentration of 0.5 ppm, with adsorbent dosages ranging from 10 to 50 mg/L and contact times from 30 to 210 minutes. Experimental results show neem powder achieving the highest removal efficiency (up to 99.57%) with residual fluoride levels as low as 0.058 ppm. Isotherm analysis showed the best fit for neem in Langmuir (R^2 = 96.72%), Freundlich and Temkin (R^2 = 99.20%) models. Kinetic studies revealed pseudo-second order as the best fit, especially for orange and amla (R^2 = 99.80%). The novelty lies in comparing low-cost, eco-friendly fruit-based bioadsorbents under identical conditions, establishing neem as the most promising for defluoridation.

Keywords: *Fluoride ions, Water, Multiple Bioadsorbents, Neural Networks, Adsorption Isotherms, pH Value*

1. Introduction

Pollutants are the substances that contribute to environmental pollution. It exist in various forms [1]. Historically, air, water, and soil were pure, supporting healthy ecosystems. However, human activities have led to significant pollution and environmental harm [2]. Advancements in science and technology have rapidly increased industries, leading to significant environmental pollution and ecological imbalances [3]. People's habits have altered as pollution levels have increased. 71% of the Earth's surface is made up of water, which is essential to all life [4]. Approximately 2% of this water is locked in the Antarctic Ice Sheet, while the remaining 97% is found in seas. In rivers, lakes, and aquifers, just 1% is available as freshwater [5]. The world's water supplies have increased significantly during the past three

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centuries. Approximately 3240 cubic kilometres of freshwater are used annually, of which 69% is used for agriculture, 23% for industry, and 8% for residential usage [6-7]. Figure 1 illustrates the contaminant levels in mg/L as per various standards. Fluorine is a reactive element in the Earth's crust, forming fluorides with most elements except noble gases [8]. It occurs in minerals like fluorapatite and fluorite and can leach into soil and water. Industrial activities, particularly aluminum production and fertilizer manufacturing, contribute to fluoride pollution, while volcanic sources can also create high concentrations [9-10]. Excessive fluoride exposure poses health risks, including skeletal and dental fluorosis, increased fracture risk, and potential cancer links [11-12]. In India, over 11 million people in 160 districts are affected by fluorosis due to water fluoride levels ranging from 1.5 mg/L to 16 mg/L, leading to discoloured teeth and severe joint issues [13-16].

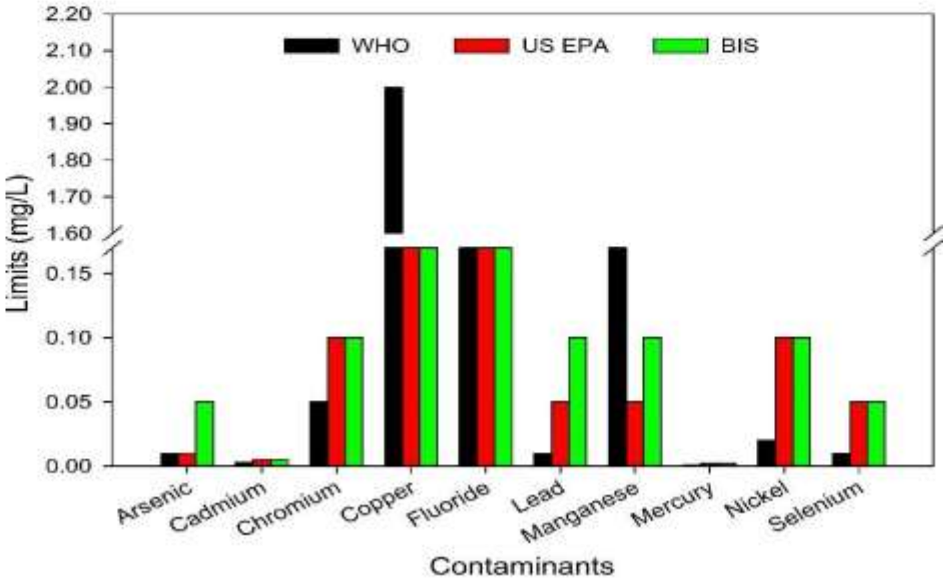


Figure 1: Contaminants and their limits

In most countries, research is still being conducted to study the effects of fluoride ions and their impact on human health. Kumar and Chawla discovered that fluoride contamination from carbon-based compounds leads to health issues due to leaching of fluoride-rich rocks, and current removal methods are often costly or complex [1]. Similarly, Mokhtar et al. tested low-cost adsorbents like banana crust meal, biochar, and wasted tea leaves for pesticide removal from polluted water. Results showed banana crust meal effectively removed pesticides like Atrazine, Diurin, Chloropyrifos, Dimethoate, and Imidacloprid [3]. Darren et al. studied seawater desalination using nanofiltration modelling methodologies, emphasizing the need for thorough membrane modelling for multi-component and highly concentrated salt solutions [5]. Chakrabortty et al. utilized cross-flow nanofiltration to study transport modelling and economic assessment of fluoride ion-polluted groundwater. The study found that this method removed fluoride up to 98% and significantly reduced the high pH value of polluted groundwater, suggesting membrane technology as a low-cost solution [6]. Mohammad et al. analysed literature on nanofiltration membranes, which were first used in the late 1980s for polluted water, wastewater, and desalination processes. They explored developments, transport mechanisms, interfacial polymerization, additives, UV grafting, electron beam irradiation, plasma treatment, and layer-by-layer modification [18]. Mondal et al. used aluminium-impregnated coconut fibre ash (AICFA) to remove fluoride from a fluoride solution with natural water. The adsorption process was spontaneous, practicable, and exothermic, eliminating 98% of fluoride with a pH value of 12 [8]. Rafique et al. conducted a study on fluoride removal from drinking water using modified immobilised activated alumina (MIAA). MIAA outperformed IAA by 1.35 times, with over 90% fluoride elimination within an hour. Compared to activated charcoal, MIAA had 10% greater efficiency and a regression coefficient of 0.99 [9]. Mourabet et al. utilized surface response methodology (RSM) to study fluoride removal from aqueous solutions using hydroxyapatite as an adsorbent. They optimized process parameters, finding the best results for temperature, pH, dose, and fluoride concentration with an accuracy of 86.34%. The study also found hydroxyapatite strongly associated with Langmuir and Freundlich models [10]. Farah et al. used white-rot fungus to remove fluoride from aqueous solutions using various factors. The Langmuir model was found to be the most accurate, and the adsorption process followed the pseudo-second-order model. The study demonstrated the low-cost, environmentally friendly, and effective method [11]. Rao and Raj study on bleaching powder's effectiveness in defluoridating water found that longer contact periods and higher doses improved removal efficiency, with neutral pH having 64% effectiveness and alkaline pH 52% [12].

Zhongping Li et al. synthesized a luminous conjugate microporous polymer (BCMP-3) for fluoride ion removal using Triarylboron-linked conjugated microporous polymers. The polymer functions as a colorimetric and fluorescent chemosensor with good fluoride sensitivity and selectivity. It can also be used as an adsorbent to remove fluoride from water, with good properties and reusability [13]. Wilson et al. studied fluoride temperature dependence and thermodynamic adsorption using activated coconut shell carbon, montmorillonite, and rice husk ash. They found that fluoride removal effectiveness improves with temperature, with activated coconut shell carbon having the highest efficiency at 83.5%. The study found exothermic, random, and spontaneous processes [13]. Katarzyna et al. conducted a study using batch electrolysis to remove fluoride ions from aqueous solutions. They found that decreasing fluoride concentration improved separation efficiency, and the predicted electrical energy requirement varied with fluoride solution content [15]. Gayathri et al. conducted a study on groundwater defluoridation using low-cost adsorbents like amla powder, coconut shell powder, neem powder, and turmeric powder, finding amla powder more effective and cost-effective [16]. Singh et al. found that calcium ions effectively reduce fluoride levels in India's contaminated groundwater, which can exceed 30 mg/L and lead to serious health issues like skeletal fluorosis from long-term consumption [17]. Nemade et al. studied water defluoridation using low-cost adsorbents to address fluoride poisoning in drinking water. They found that fishbone charcoal is more efficient and cost-effective than other types of charcoal, highlighting the need for effective solutions [18]. Zuoze et al. studied water defluoridation using nonwoven silk fibroin and polypropylene textiles as adsorbents. They created nanofibers with large surface areas and fluoride affinity, which formed crystals and increased in diameter with increasing SF concentration. Fluoride was eliminated within 20 minutes [18]. Chidambaram et al. conducted an experiment to eliminate fluoride from water using natural materials. They used five bio adsorbents: charcoal, fly ash, red soil, brick, and serpentine. Red soil was found to be the most effective in removing fluoride ions, followed by brick, fly ash, serpentine, and charcoal [19]. Margandan et al. conducted a literature review on groundwater defluoridation in India, focusing on high fluoride concentrations in dry and semiarid zones. They examined various procedures, their applications, and pros and cons [20]. Tesfaye et al. conducted a study on sorption technology for removing fluoride from water using diatomite modified with aluminium hydroxide. The study found optimal parameters for dosage, contact duration, starting fluoride concentration, and pH value, with a maximum fluoride removal efficiency of 89.4%, using pseudo-second-order kinetics [21]. Beraki et al. conducted an experiment in Keren, Eritrea, using various sorbent materials like domestic ash, crushed burned clay pot, Keren, and adigerghish soil for water defluoridation. The study found that crushed burned clay pots had the highest pH value and eliminated 0.26 mg fluoride/g medium, while domestic ash had the lowest. A literature review examined various adsorbents for fluoride removal from water [22]. Parlikar et al. found that acid-treated tea ash powder was more effective than alkali-treated versions, achieving optimal results at 400 mg/L concentration and 150 minutes of contact time [23].

Other studies explored materials such as bottom ash (Ramesh et al.), banana peel and passion fruit (Saranya et al.), moringa oleifera and tulsi powder (Aleena et al.), neem stem charcoal (Chakraborty et al.), and brick powder (multiple authors). Nair et al. focused on composite beds, while Kiran et al. evaluated activated carbon from lemon peels. Additionally, Chavan et al. used beetroot and okra seeds, and Gandhi et al. tested various materials including chalk and seed powders. Overall, fluoride removal efficiency ranged from 56.1% to 94%, with pH values between 2 and 10 and contact times of 60 to 210 minutes [24-26]. Several authors have explored the defluoridation of water using various low-cost adsorbents. Notable examples include Bhaumik et al. with eggshell powder, Gourouza et al. using beef bones, and Goswami et al. employing neem leaf powder. Other studies by Chakrapani et al. investigated activated carbon, while Patil et al. analysed a range of plant powders. Additionally, Aashmohammed et al. utilized banana peel and groundnut shell, and Dwivedi et al. focused on citrus limetta and peepal leaf powder. Harikumar et al. examined vetiver root, tamarind seed, and clove, while Sudarshan et al. worked with tulsi leaves powder, and Chakraborty et al. used neem charcoal powder. Collectively, these studies demonstrate that these adsorbents are effective in removing fluoride from various water sources [27-29]. The literature review highlights the severe health risks associated with elevated fluoride levels in water and the growing demand for effective remedies. Numerous studies have shown that natural, inexpensive, and modified adsorbents offer encouraging fluoride removal outcomes. Cutting-edge techniques, such as membrane technology and nanofiltration, also demonstrate high efficiency and cost-effectiveness. All of these results underscore the importance of employing flexible and sustainable defluoridation methods.

2. Experimental Program

The experimentation was performed in batches using a constant residual concentration of 0.5 ppm with adsorbent dosages ranging from 10 to 50 mg/L and contact times of 30 to 210 min, in increments of 10 mg/L and 30 min, respectively. Prior to experimentation, the reactivity of metal compounds, including iron, aluminum, thorium, zirconium, lanthanum, and cerium, with an indicator dye to form a stable complex, was examined using a spectrophotometric approach. A spectrophotometer can detect the structural shift in the absorption spectrum caused by this complex's reaction with fluoride. Standard NaF fluoride stock solution, SPANDAS solution, zirconyl acid solution, and acid-zirconyl-SPANDAS reagent were among the reagents made. During an experiment, three bioadsorbents-Neem powder, Amla powder, and Orange powder-were utilized. Furthermore, the pH value was measured with the help of a digital pH meter made by Decibel DB1011. With the help of TOPSIS, a multi-criteria decision-making technique (MCDM), the performance of the adsorbents has been evaluated and based on the data. The adsorption isotherms such as Langmuir, Freundlich, and Temkin isotherms were calculated followed by pseudo first and second order kinetic models.

3. Adsorption Isotherms

Fluoride's accumulation on an adsorbent surface at constant temperature allows for the creation of an adsorption isotherm. Common isotherm models for analyzing adsorption data include linear models and non-linear models such as Langmuir, Freundlich, Dubinin-Radushkevich, Redlich-Peterson, Elovich, and Temkin isotherms [30-32]. The current study mainly focussed on Langmuir, Freundlich, and Temkin isotherms. The Langmuir model evaluates the adsorbent's maximum fluoride ion absorption capacity based on monolayer coverage and homogeneous surface uniformity, calculating maximum adsorption capacity through linear and non-linear data fitting.

$$q_e = \frac{q_m b C_e}{1+bC_e} \quad (1)$$

The Freundlich isotherm evaluates linear and non-linear data on fluoride adsorption on heterogeneous bioadsorbent surfaces, revealing multilayer, cooperative, and monolayer or L-type adsorption due to physical or chemical interrelations.

$$q_e = K_F \times C_e^{\frac{1}{n}} \quad (2)$$

The Temkin isotherm indicates that the heat of all molecules decreases with an increase in surface coverage, as expressed as,

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (3)$$

The adsorption kinetics were studied using linear pseudo first and second order equations in line with the adsorption isotherm [33-34].

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_t} \quad (5)$$

The non-linear equations of pseudo first and second orders, which can be expressed as,

$$q_t = q_e(1 - e^{-k_1 t}) \quad (6)$$

$$q_t = \frac{k_2 q_e^2}{1+k_2 q_e t} \quad (7)$$

k_1= Rate constant for pseudo first order

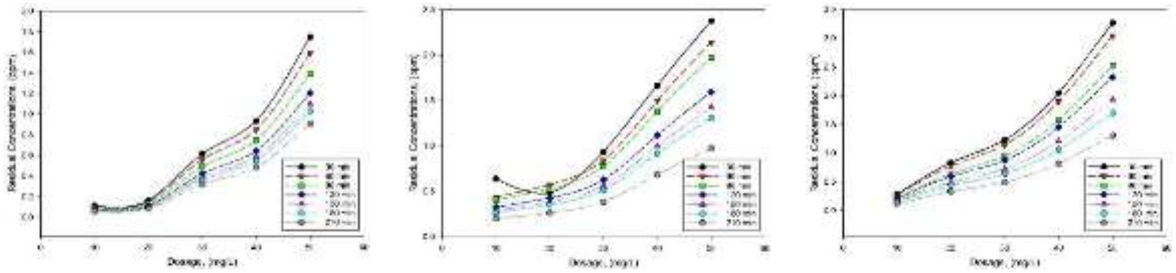
k_2= Overall rate constant for pseudo second order

The adsorption kinetics can also expressed by using Weber-Morris inter particle diffusion,

$$q_t = k_{int} \times t^{0.5} \tag{8}$$

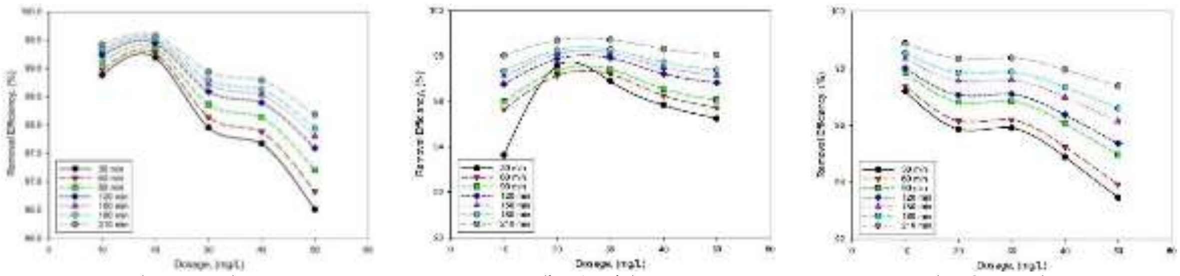
4. Results and Discussion

The results are presented based on the experimental data. The graphs are plotted for residual concentrations, removal efficiency, and pH values against the adsorbent dosages for 0.5 ppm fluoride concentrations. Figure 3 illustrates the adsorbent dosages versus residual concentrations. An increasing trend is observed in all the bioadsorbents under considerations. Residual concentration observed for neem (3a) in the range of 0.058-1.748 ppm, amla (3b) shows 0.198-2.377 ppm, and orange (3c) shows 0.112-3.269 ppm. Highest and lowest residual concentrations were observed at 30 min and 210 min contact timings for all the adsorbents under considerations. Figure 4 illustrates the removal efficiency for various bioadsorbents under considerations. The removal efficiency is observed in the range of 96.50%-99.57% for (4a) neem adsorbent and 93.64%-98.72% for (4b) amla, and 93.46%-98.87% for orange (4c) powder adsorbents. Highest and lowest efficiencies are observed for all the bioadsorbents under considerations at 210 min and 30 min contact timings.



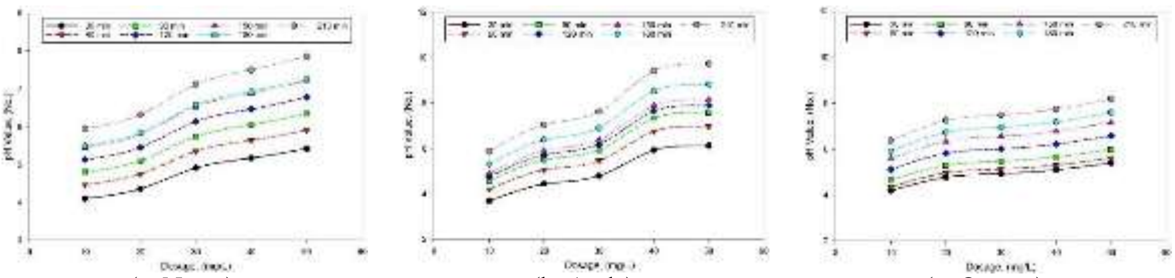
(a: Neem) (b: Amla) (c: Orange)

Figure 3: Adsorbent dosages versus residual concentration



(a: Neem) (b: Amla) (c: Orange)

Figure 4: Adsorbent dosages versus removal efficiency



(a: Neem) (b: Amla) (c: Orange)

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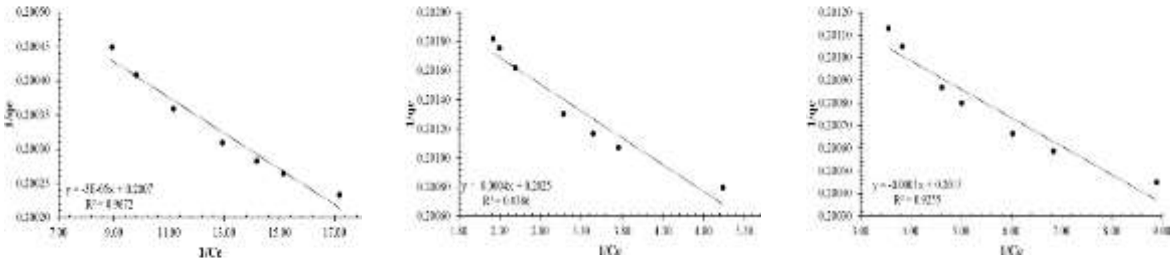
Figure 5: Adsorbent dosages versus pH value

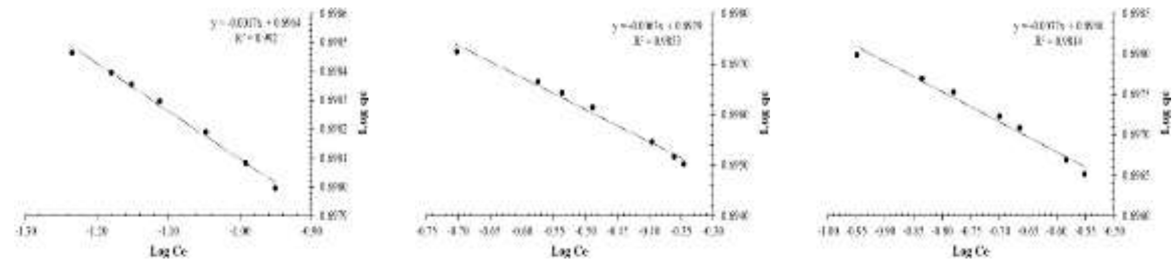
Figure 5 illustrates the pH values for various bioadsorbents. The pH values are observed within the range of 4.10-7.86 for (5a) neem powder, 3.69-9.73 for (5b) amla powder, 4.19-8.19 for (5c) orange powder adsorbents. The pH values increases with increase in adsorbent dosages and contact timings. The optimal, acidic and alkaline nature of pH values are observed during experimentation. As the dosage and contact time of neem, amla, and orange powder adsorbents increase, basic functional groups like hydroxyl and carboxyl are released, which neutralize acidic substances in the water, resulting in a gradual increase in pH during fluoride removal.

A batch experimentation was performed on defluoridation of water on constant fluoride concentrations of 0.5 ppm with adsorbent dosages ranging from 10 to 50 mg/L and contact timings of 30 to 210 minutes. The study utilizes isotherms like Langmuir, Freundlich, and Temkin to analyze experimental data, enhancing understanding of adsorbent behaviour, liquid phase distribution, and surface properties. The graphs are plotted for various isotherms under considerations for different bioadsorbents.



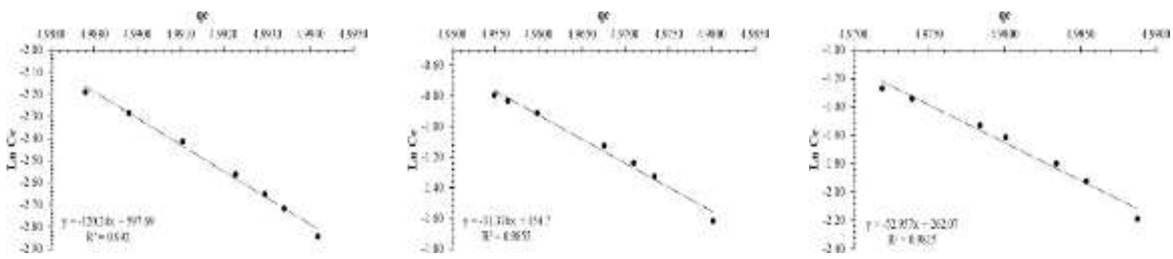
(a: Neem) (b: Amla) (c: Orange)

Figure 6: Langmuir isotherms for bioadsorbents



(a: Neem) (b: Amla) (c: Orange)

Figure 7: Freundlich isotherms for bioadsorbents



(a: Neem) (b: Amla) (c: Orange)

Figure 8: Temkin linear isotherms for bioadsorbents

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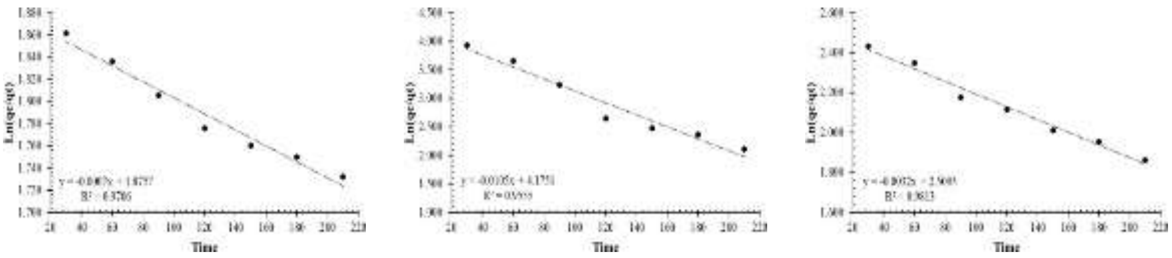
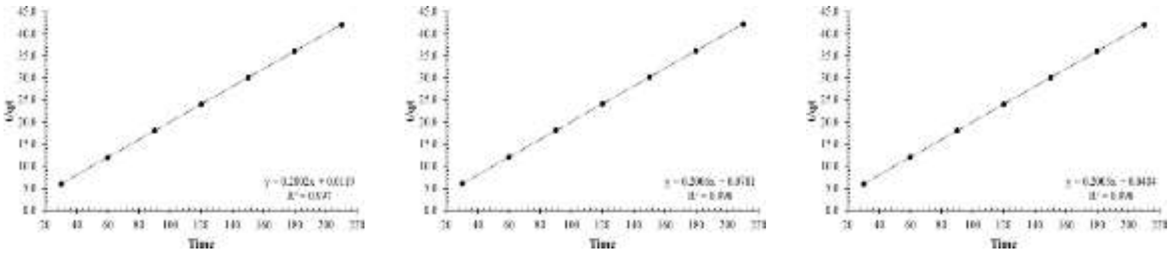
Figure 6 illustrates the Langmuir isotherms for adsorbents under considerations. From the plotted graph $1/C_e$ versus $1/q_e$ it is clearly observed that highest R^2=96.72% for (6a) neem adsorbent followed by 93.86% and 92.55% for (6b) amla and (6c) orange powder adsorbents respectively. Similarly Figure 7 illustrates the Freundlich isotherm for bioadsorbents under considerations. The graphs plotted for Log C_e versus Log q_e shows that highest R^2= 99.20% for (7a) neem powder adsorbent followed by 98.53% and 98.14% for (7b) amla and (7c) orange powder adsorbents respectively. Figure 8 illustrates that Temkin isotherms for various bioadsorbents under considerations. The graphs plotted for q_e versus Ln C_e shows that R^2=99.20 for (8a) neem powder adsorbent followed by 98.55% and 98.15% for (8b) amla and (8c) orange powder adsorbents respectively. Among the bioadsorbents studied, neem powder showed the highest adsorption efficiency with the best fit across all isotherm models, evidenced by the highest R^2 values in Langmuir (96.72%), Freundlich (99.20%), and Temkin (99.20%) plots. This confirms neem as the most effective adsorbent compared to amla and orange powders.



(a: Neem) (b: Amla) (c: Orange)

Figure 9: Pseudo-first order kinetic model for bioadsorbents



(a: Neem) (b: Amla) (c: Orange)

Figure 10: Pseudo-second order kinetic model for bioadsorbents

The study uses regression analysis to analyze linear kinetic models for adsorption using multiple adsorbents under considerations for evaluating equilibrium time. Figure 9 illustrates the pseudo-first order kinetic models for different bioadsorbents such as a: Neem, b: Amla, and c: Orange. In pseudo-first order kinetic model the highest R^2 observed 98.13% for (9c) orange powder adsorbent followed by 97.06% and 95.55% for (9a) Neem and (9b) Amla powder adsorbents respectively. Figure 10 illustrates the pseudo-second order kinetic model for different bioadsorbents under considerations. The pseudo-second order model predicts the R^2

with an accuracy of 99.80% for (10c and 10b) orange and amla powder adsorbents whereas neem powder (10a) showed 99.70% accuracy. The pseudo-second order model best describes the adsorption kinetics, with orange and amla powders showing the highest R^2 of 99.80%. This suggests chemisorption is the dominant mechanism for these bioadsorbents.

5. Conclusions

The conclusions are drawn based on the experimental and numerical investigations:

- Neem powder demonstrated the highest fluoride removal efficiency, with residual concentrations ranging from 0.058 to 1.748 ppm, outperforming amla and orange powders.
- Removal efficiency increased with contact time, reaching up to 99.57% for neem, 98.87% for orange, and 98.72% for amla at 210 minutes.
- pH values increased with both adsorbent dosage and contact time due to the release of basic functional groups from the bioadsorbents, influencing the water's alkalinity.
- All three bioadsorbents-neem, amla, and orange showed effective fluoride removal at 0.5 ppm concentration, with neem powder being the most efficient.
- Langmuir isotherm analysis revealed strong monolayer adsorption, with neem powder exhibiting the highest R^2 value of 96.72%, followed by amla and orange.
- Freundlich and Temkin's isotherms further confirmed neem's superior performance with the highest R^2 values of 99.20%, indicating heterogeneous surface adsorption and adsorbate-absorbent interaction.
- Pseudo-first-order kinetics showed the best fit for orange powder (R^2 = 98.13%), indicating a partial physical adsorption process.
- Pseudo-second order kinetics best described the adsorption mechanism, with orange and amla powders showing the highest R^2 values (99.80%), indicating chemisorption as the dominant process.
- Isotherm and kinetic models validated the experimental data, helping to understand the adsorption behaviour and mechanisms involved in fluoride removal.
- Overall, neem powder emerged as the most promising bioadsorbent across all isotherm and kinetic models, followed closely by orange and amla powders, making them suitable for eco-friendly water defluoridation applications.

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Abbreviations

Abbreviations	Full-form
q_m	Maximum adsorption capacity, mg/g
b	Constant related to energy adsorption
C_e	Equilibrium concentration of fluoride in mg/L
K_F	Adsorption capacity
n	Adsorption intensity
B_1 and K_T	Estimated from plotting q_e and C_e
%	Percentage
mg	Milligram
L	Litre