



Exploring the Adsorption Efficiency of Local Apricot Seed Shell as a Sustainable Sorbent for Nitrate Ion

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ABSTRACT

Locally available apricot seed shell as agro-waste was used for the preparation of adsorbents. The biochar was prepared at 370°C via pyrolysis and 80 mesh particle sizes were modified by 1N HCl. Nitrate adsorption and effect of co-ions from aqueous solution were studied under batch model using apricot seed shell powder (ASSP), apricot seed shell biochar (ASSB), and activated apricot seed shell biochar (AASSB). FTIR and pH_{PZC} measurements were used to characterize the adsorbents. Based on the experimental findings, the optimum conditions follow pH 2, 0.3g dosage, initial concentration of 50 mg.L⁻¹, and contact time of 90 min. The three forms of adsorbent exhibited good adsorption for nitrate. However, the maximum percentage removal of nitrate ions from the aqueous solution followed the order AASSB>ASSB>ASSP. The adsorption kinetic of nitrate ion was best fitted by pseudo 2nd order, and the parameters of adsorption isotherms elucidated favorable and improved sorption. This agro-waste could be used to develop sustainable adsorbents in water and wastewater treatment methods and has great potential to replace commercially available sorbents.

INTRODUCTION

Worldwide, nitrate is a common pollutant of surface and groundwater. Water nitrate pollution is a serious problem, especially in arid and semi-arid areas (Shukla & Saxena 2020). Nitrate is a polyatomic ion, stable, and highly soluble in water. It is frequently caused by runoff from farms, poorly maintained residential and commercial sewage systems, massive trash dumps, atmospheric fixation, and lightning (Shukla & Saxena 2020, Zhou 2015). In 1945, nitrate toxicity was identified for the first time (Zhou 2015). Nitrate poisoning in humans can occur through two different routes, including oral exposure and cutaneous contact. However, oral exposure is more common than dermal contact in the general population (Rahman et al. 2021). World Health Organization (WHO) has set a recommended threshold value of 50 mg.L⁻¹ for nitrate in drinking water, considering the health hazards, likely the possibility of methemoglobinemia or “blue baby syndrome” in young children linked to elevated levels of nitrate in the water (Shukla & Saxena 2020c, Zhou 2015c). This disorder arises from the body’s conversion of nitrates found in water to nitrites, which can disrupt hemoglobin’s ability to deliver oxygen throughout the blood. Thus, in order to address this issue, appropriate management and mitigation techniques are essential. There are numerous

ways and strategies available to remove different types of contaminants from water (Dai et al. 2018, Khatoon & Rai 2016, Sardar et al. 2021). The adsorption approach is regarded as the most effective treatment method owing to its affordability, simplicity, high absorption capabilities, and diversity among all other techniques (Dai et al. 2018, Das et al. 2020, Mitra et al. 2019).

A large volume of agricultural waste is produced during harvesting, post-harvesting, and processing, which can be converted into various useful products (Tiwari & Pal 2021). The agricultural biomasses, such as the shell or peel from fruits and vegetables, sugarcane bagasse, rice husks, peanut shells, jackfruit, chestnut shells, coconuts, and hazelnuts, were used to remove a range of contaminants (Khatoon & Rai 2016). According to published research works, the underlying chemical structure and composition of raw agricultural biomasses have been shown to have a somewhat lower capacity for the adsorption of pollutants (Liu et al. 2022). As a result, surface alterations were suggested for improved adsorption capacities. Nitrate was reduced by using powdered banana peel (Reddy et al. 2015). While banana peel biochar was used for the reduction of nitrate (Zounggran et al. 2021). Nitrate was eliminated using barley straw (Ansari et al. 2017). Biochar is a promising material



Fig. 1: Systematic presentation of biochar preparation.

for treating water. It is made by pyrolyzing, gasifying, and hydrothermally carbonizing at high temperatures (between 300-900 °C) in an inert atmosphere (Janković et al. 2019).

Worldwide, approximately 3.72 million tonnes of apricots are produced. The main producers of apricots are Turkey, Uzbekistan, Iran, Italy, and Algeria. Ladakh is the largest apricot-producing state in India. Himachal Pradesh and Jammu and Kashmir also produce it. Every year, Ladakh produces 15000 metric tons of apricots. According to the Horticulture Department, Kargil UT Ladakh, an estimated 1655 hectares of land is under apricot production in district Kargil of UT Ladakh, with an annual production of 11,067.46 Metric tons. During the winter, the seed shell is typically burned in the local heating system to warm the rooms in this region. The shell is hard, and agro-waste is produced during the kernel separation process.

Moreover, a substantial amount of apricot seed shells is produced as agro-waste during the processing of fruits worldwide, which are disposed of either as agro-waste or used to improve soil fertility (Hashem et al. 2022, Challab et al. 2020, Zhang et al. 2022a). The different forms of apricot seed shell including powder, biochar, and activated carbons, were utilized by different researchers for the removal of pollutants. The powder form of apricot seed shell was used to remove acid blue 193 dye (Hashem et al. 2022), activated carbon was used for the removal of metals like Mn^{2+} and Zn^{2+} , and similarly, biochar of this material was used for the removal of pesticides like atrazine (Zhang et al. 2022b). In this work, biochar and activated biochar from locally available apricot seed shells were prepared, and nitrate sorption behavior was assessed.

MATERIALS AND METHODS

Chemicals and Materials

Apricot seed shell was collected from the oil processing unit at Hardas, Kargil of UT Ladakh. All the chemicals of analytical grade, such as potassium nitrate (KNO_3), hydrochloric acid (HCl), sodium hydroxide (NaOH), and other reagents were used. Deionized water was used for preparing the solution, and synthetic water was used.

Preparation of Adsorbents and Synthetic Water

Apricot seed shells were washed with water and deionized water to remove dirt. The dry seed shells were then crushed to a fine powder and sieved using 80 mesh sieves. The apricot seed shell powder (ASSP) was then preserved in an airtight container for further application. 300g of crushed apricot seed shells were placed in the electric furnace reactor and heated for 5 h with a continuous flow of nitrogen gas at temperatures of 300°C, 330°C, 350°C, and 370°C. The resulting apricot seed shell biochar (ASSB) is then passed through an 80-mesh sieve. The biochar was then washed with deionized water until neutral pH was reached and then left to dry at 50°C. Fig. 1 systematically shows the biochar preparation process.

The impact of temperature on the biochar yield during pyrolysis is summarized in Table 1. Synthetic water of 100 ppm, 70 ppm, 50 ppm, 30 ppm, and 10 ppm was prepared by dissolving potassium nitrate (KNO_3) in deionized water. Synthetic water containing nitrate and other ions like fluoride, chloride, Phosphate, and sulfate was also prepared to investigate the interfering ions on the adsorption efficiency of nitrate ions.

Modification of adsorbent: The apricot seed shell biochar (ASSB) was modified by using a 1N HCl solution. The 10g of ASSB was soaked in 1 N HCl solution for 5 h and then separated the biochar. The biochar was washed with water till the pH of the filtrate became 7. The biochar was allowed to dry in the oven at 60°C, and acid-modified apricot seed shell biochar (AMASSB) was preserved for further use.

Characterization of biochar: The surface functional groups and surface charge of ASSP, ASSB, and AASSB were investigated by using a Fourier transform infrared (FTIR) spectrometer and zero point charge (pHpzc).

Table 1: Effect of working temperature on yield of Biochar.

| Temperature [°C] | Yield [g] |
|------------------|-----------|
| 330 | 210.14 |
| 330 | 222.67 |
| 350 | 258.64 |
| 370 | 209.337 |

Determination of pH at zero point (pH_{pzc}): The pH_{pzc} is the pH at which the net surface charge of a given sorbent is zero. It is an important parameter to determine the extent of uptake capacity of charged ions by the sorbent. The pH of the solution greater than pH_{pzc} favors the adsorption of cationic ions, while pH less than pH_{pzc} favors the adsorption of anionic ions (Hashem et al. 2022). Ten samples of varying pH (2-10) were prepared by using 0.01 M NaCl as a base electrolyte in a volumetric flask, and the pH of the solution was adjusted by using 0.1N HCl and 0.1N NaOH solution. 1g of the biochar is added in 50 mL of each flask and kept in the shaker for 6h and recorded pH using a pH meter as pH_{final}. A plot of change in pH (pH_{final}- pH_{initial}) against pH_{initial} was generated. The pH_{pzc} of the adsorbents was recorded where ΔpH was zero.

Batch adsorption analysis: A stock solution of 100 ppm nitrate solution was prepared and diluted to form 70 ppm, 50 ppm, 30 ppm, and 10 ppm nitrate solutions. 10 mL of each solution was taken in a beaker and evaporated to dryness on a hot plate. Added to each 2 mL of phenoldisulphonic acid (PDA) and then added 10mL ammonia solution. A yellow solution was obtained due to rearrangement in the structure of the nitro derivative. Absorbances of the series of solutions were recorded at 410 nm wavelength using a UV-visible spectrophotometer, and the calibration curve between absorbance and concentration was plotted. To investigate the adsorption, 0.3g of biochar was placed in a centrifuge tube, and 50 mL nitrate solution was added. After centrifugation at 200 rpm, the supernatant was taken at the contact time of 10, 30, 50, 70, 90, 120,140, and 160 min. After filtration through Whatman grade 1 filter paper, the same procedures were applied, and determined the concentrations were from the calibration curve.

Influencing Variables on Adsorption

Effect of pH: The effect of solution pH on the adsorption of nitrate was investigated by taking 50 mL of nitrate solution and 0.3g of adsorbents. The pH of the solutions was adjusted to 2, 4, 6, 8, and 10 with 0.1 N solutions of HCl and NaOH. The content of nitrate ion after 90 min of contact time was determined in the supernatant.

Effect of contact time: The effect of contact time on the adsorption of nitrate ion on the adsorbents was determined by taking a 50 mg.L⁻¹ initial concentration of nitrate at 2 pH by using 0.3 g of adsorbents. The nitrate concentrations were determined at the interval of 10, 30, 50, 70, 90, 120, 140 and 160 min.

Effect of dosage: The effect of the dosage of adsorbent was investigated by using 50 mg.L⁻¹ of nitrate solution by using dosages of 100, 200, 300, 500, and 700 mg of adsorbents at the contact time of 90 min and 2 pH.

Effect of initial concentration: The effect of the initial concentration of nitrate solution on the adsorption was determined by taking 10, 30, 50, 70, and 100 mg.L⁻¹ of nitrate solutions by using the dosage of 300 mg.

Effect of co-ions: To determine the effect of co-ions on the adsorption of nitrate, 10 mg.L⁻¹ of fluoride, chloride, phosphate, and sulphate were used. 10 mL of each co-ion solution was added one by one to the nitrate solutions, and the concentration before and after the addition of co-ion was determined.

Data Processing

MS Excel was used to calculate the various parameters involved in adsorption kinetics and isotherms. MS Excel 2010 was used to analyze the experimental data and to draw the figures.

Calculation of adsorption capacity and removal percentage: The amount of nitrate ion adsorbed (mg.g⁻¹) by the sorbents was calculated by using the following formula

$$Q_e = (C_i - C_e) V/m \quad \dots(1)$$

$$Q_t = (C_i - C_t) V/m \quad \dots(2)$$

The percentage removals of nitrate by the sorbents were calculated by the following formula.

$$\% \text{ Removal} = (C_i - C_e) / C_i \times 100 \quad \dots(3)$$

Where Q_e is the adsorption capacity at equilibrium (mg.g⁻¹), Q_t is the adsorption capacity at time t. The initial concentration (C_i) of adsorbate (mg.L⁻¹) and C_e is the concentration after time t. V is the Volume (50 mL), and m is the mass of the adsorbent (g).

Adsorption kinetics: The adsorptions of nitrate ion on the sorbents were fitted to pseudo 1st order and pseudo 2nd order kinetic models.

The equation for Pseudo first-order kinetic model:

$$\ln (Q_e - Q_t) = \ln Q_e - K_1 t \quad \dots(4)$$

The equation for Pseudo first-order kinetic model:

$$t / Q_e = 1 / K_2 Q_e^2 + 1 / Q_e \quad \dots(5)$$

Where Q_t and Q_e are the adsorption capacity at time t and equilibrium, K₁ and K₂ are the rate constant for pseudo 1st order and pseudo 2nd order models.

Adsorption isotherm: The adsorption of nitrate ion onto the various forms of adsorbents of apricot seed shell was fitted to the Langmuir and Freundlich model.

Langmuir model:

$$1 / Q_e = 1 / K_L Q_{max} + 1 / C_e + 1 / Q_{max} \quad \dots(6)$$

Freundlich model:

$$\log Q_e = \log K_f + 1/n \log C_e \quad \dots(7)$$

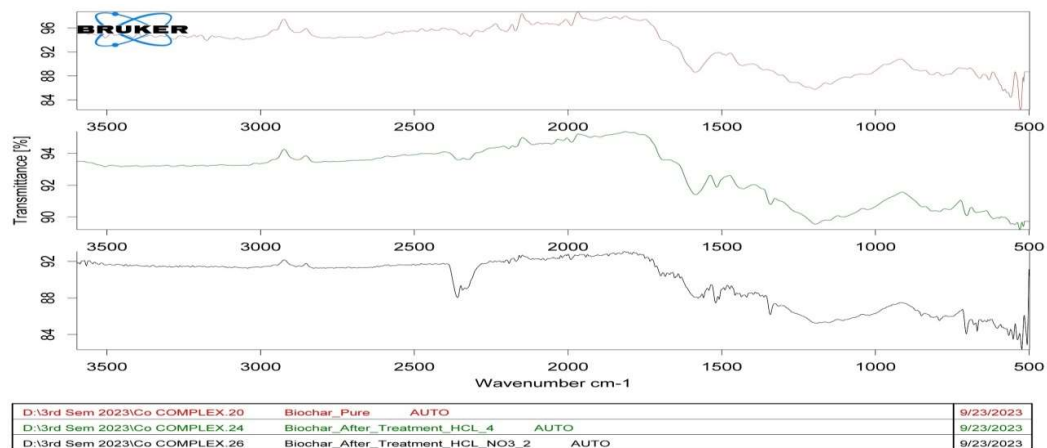


Fig. 2: FTIR Spectra.

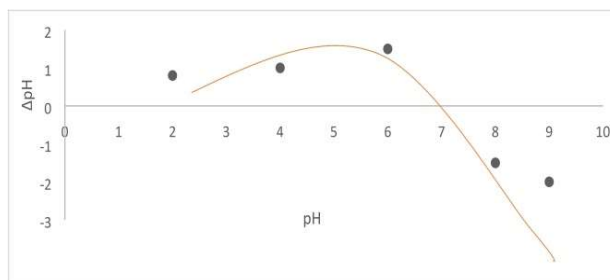


Fig. 3: Point of Zero charge (biochar).

Where Q_e is the adsorption capacity of the adsorbent at equilibrium, K_L and K_f are the Langmuir and Freundlich isotherm constant, Q_{max} is the maximum adsorption capacity ($\text{mg}\cdot\text{g}^{-1}$), and C_e is the equilibrium concentration ($\text{mg}\cdot\text{L}^{-1}$).

RESULTS AND DISCUSSION

Characterization of Adsorbents

Fourier transmission infrared spectrometric analysis (FTIR):

The IR spectra of Apricot seed shell biochar (ASSB), acid treated, and nitrate loaded is shown in Fig. 2. A prominent peak around $1650\text{--}1700\text{ cm}^{-1}$ indicated the presence of an unsaturated carbonyl ($\text{C}=\text{O}$) group. There are no prominent changes in the spectra on treatment with acid. However, loading with nitrate, a shift in the spectra was seen around 2400 cm^{-1} , which is probably due to the $\text{O}=\text{C}=\text{O}$ bond, and a band around 1550 cm^{-1} probably due to the $\text{N}-\text{O}$ bond. These bands revealed that there is the formation of a bond between the surface functional group and NO_3 .

The pH point of zero charge (pH_{PZC}) analysis: The adsorption capacity of the adsorbent depends on the point of zero charge (Hashem et al. 2022). The pH point of zero

charge (pH_{PZC}) of the adsorbent was determined and found to be around pH 7.9, 7.4, and 8.9 for ASSP, ASSB, and AASSB. The plot is shown in Fig. 3.

Effect of Variables on Adsorption

Effect of pH: Solution pH is an important factor that controls the adsorption process. The powder and biochar form of apricot seed shell showed very low absorption capacity at higher pH ($\text{pH} > 7$). This indicates negatively charged surface functional groups that can cause anionic repulsion. In this study, the effect of solution pH on adsorption capacity was investigated in the range of pH 2-10 using a constant concentration of 50 ppm nitrate and 0.3 g of adsorbents. The experimental results are shown in Fig. 4(a). The results showed that a decrease in solution pH favors the adsorption, and removal percentages become maximizing at a solution with pH 2. At lower pH, the biochar surface gets positively charged, and the interaction between biochar and adsorbent is purely electrostatic (Hashem et al. 2022d, Das & Goud 2020). As the pH increases, the adsorbent surface becomes negatively charged, and the adsorption capacity decreases due to repulsion. Moreover, the pH of the solution does not have much effect on the activated biochar, and even

at neutral pH, modified biochar (AASSB) showed good removal efficiency.

Effect of contact time and initial adsorbate concentration:

The effect of contact time at a range of 0-160 min and initial concentration of nitrate in the range of 10-100 mg.L⁻¹ were investigated, and the results were presented in Fig. 4(c, d). The equilibrium adsorption was attained around a contact time of 90 min. The slope of the initial adsorptions within the range of 90 min is larger, indicating a faster sorption rate than after the 90 min in the initial stage, the nitrate molecules quickly occupying the active sites of the adsorbents. Moreover, the adsorption process is relatively faster in the case of AASSB owing to its protonated surface.

Effect of adsorbent dosage: The effect of dosage on the adsorption capacity was investigated by taking 50 mg.L⁻¹ initial concentration of Nitrate at pH 2 by varying the dosage (100-700 mg). The result is shown in Fig. 4(b). The uptake

percentage removals were maximum at the dosage of 300 mg and minimum at the dosage of 100 mg. With the increase in dosage, the active site will increase, but also, there is the possibility of agglomeration and clogging of active sites, which decrease the adsorption capacity of the adsorbent.

Effect of co-ions: To assess the adsorbents in the real sense, the effect of co-ions, which are mostly common in water, was also investigated. Fluoride and chloride ions do not have much effect on the sorption capacity of nitrate. Still, phosphate and sulfate caused an appreciable decrease in the nitrate sorption with the increase in concentration of co-ions. Being a high negative charge on sulfate, the adsorption of nitrate is much affected due to dominant competition. The effect of co-ions on the adsorption of nitrate by the apricot seed shell biochar (ASSB) is shown in Fig. 5 (a, b).

The removal percentage of adsorbents at varying pH and initial concentration was calculated to obtain the optimum

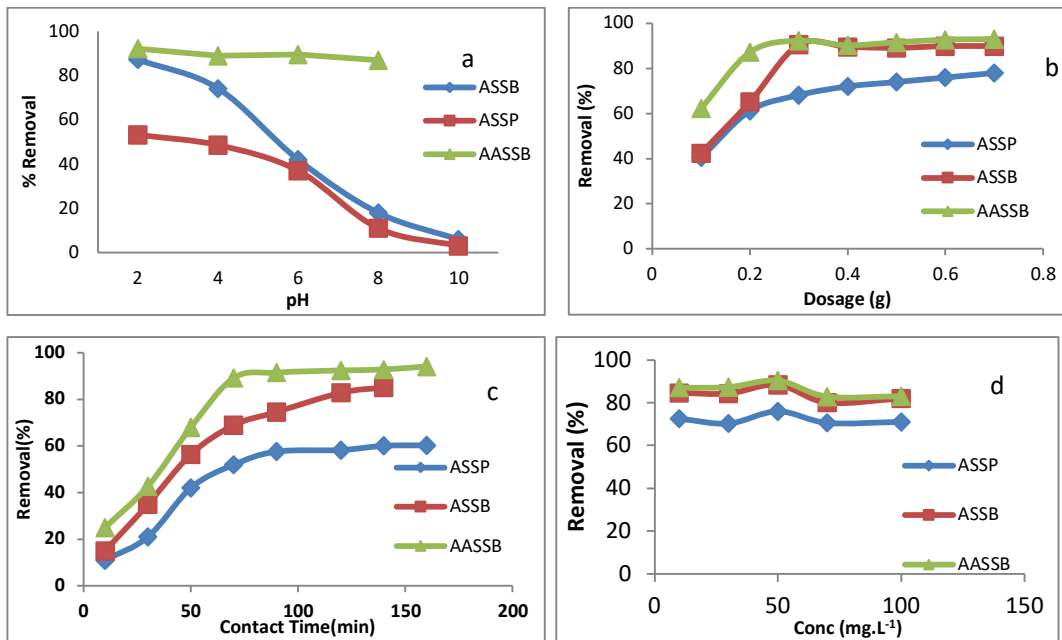


Fig. 4: Effect of variables on adsorption.

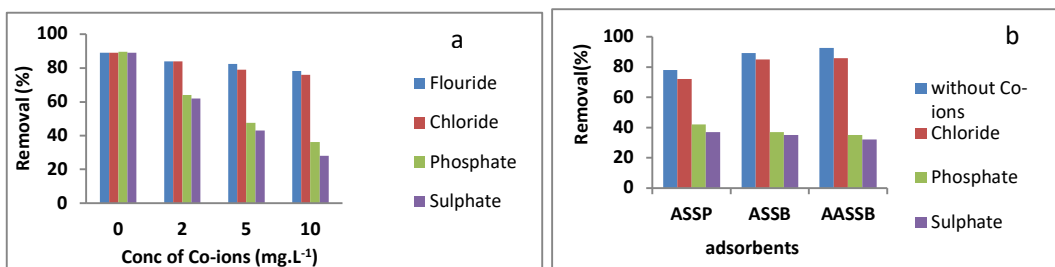


Fig. 5: Effect of co-ions.

condition. The result indicated that the maximum percentage removals was achieved at the optimum condition of 50 mg.L⁻¹ initial concentration and 0.3 g of dosage at pH 2. The higher adsorption capacity of ASSB and AASSB than the ASSP is due to greater porosity and specific surface area. The acid activation of adsorbent further enhanced the adsorption due to the protonation of functional groups on the surface of the biochar, which serves as the enhanced interaction with the negatively charged ions (Nitrate). The percentage removal of nitrate by the adsorbents of apricot seed shell followed the order- AASSB > ASSB > ASSP (Fig. 4).

Adsorption Modeling

Adsorption kinetics: The kinetic modeling is an important parameter to evaluate the rate of solute-mass transfer and the mechanism of the adsorption. In this study, pseudo, 1st and 2nd order kinetic models were applied to the adsorption of nitrate ions onto ASSP, ASSB, and AASSB. The fitting to kinetic models were evaluated and calculations of relevant parameters in Table 2 were done by using Excel 2010. The fitting parameter (R²) for the adsorption of nitrate by ASSP, ASSB, and AASSB to pseudo 1st order and pseudo 2nd order is 0.4567, 0.5894, 0.4858 and 0.9144, 0.9796, 0.9690, respectively. The adsorptions of nitrate by adsorbents were more closely fitted to pseudo 2nd order.

Adsorption isotherms: The Langmuir and Freundlich isotherm model were applied to fit the adsorption isotherm of nitrate on the adsorbents, including apricot seed shell powder (ASSP), apricot seed shell biochar (ASSB) and activated apricot seed shell biochar (AASSB). The relevant isotherm parameters are shown in Table 3. The parameters of the Langmuir isotherm model with R_L values 0 < R_L < 1 and K_L values less than 1 favor improved adsorption (Hashem

et al. 2022). The present sorption studies of nitrate on the adsorbents are efficient as the K_L and R_L values indicated favorable and improved sorption, which are attested by the larger value of Q_{max}. The values of n can elucidate the favourability and sorption affinity in terms of Freundlich isotherm- (1 < n < 10) and K_f - (K_f > 1) values (Hashem et al. 2022). The n and K_f values of the present sorption favor favorable processes.

CONCLUSION

The pollution of nitrate in water is abundant throughout the world, and research is focusing on developing strategies and technologies to prevent and reduce nitrate ions in water ecosystems. In this study, apricot seed shell powder, biochar, and acid-activated biochar have been utilized for investigating nitrate ion sorption through batch adsorption procedures. The analysis showed that the adsorption of nitrate ions onto the ASSP, ASSB, and AASSB was affected by the pH of the solution, contact time, dosage, and ion concentration. The adsorptions of nitrate ions were fitted to the Langmuir and Freundlich isotherm model, and the equilibrium parameters indicated a favorable and improved efficiency. Likewise, the adsorption of nitrate ions onto the adsorbents was simulated to pseudo 1st order and 2nd order kinetic model, and the data were analyzed using MS Excel 2010 software. The pseudo-2nd-order kinetics model well describes the coefficient of determination (R²). The equilibrium and kinetic data suggest an involvement of surface adsorption, diffusion, electrostatic, and ion exchange interaction. The adsorption efficiency of the adsorbents was greatly affected by the presence of co-ions in the solution. The percentage removal of nitrate decreases with highly negative ions like phosphate and sulfate. The apricot seed shell could be utilized for the development of effective adsorbents in water and wastewater treatment.

Table 2: Kinetic model and relevant parameters.

| Adsorbents | Pseudo 1 st Order | | | Pseudo 2 nd Order | | |
|------------|------------------------------|--------|----------------|------------------------------|---------|----------------|
| | Qe [mg.g ⁻¹] | K1 | R ² | Qe [mg.g ⁻¹] | K2 | R ² |
| ASSP | 7.821 | 0.0096 | 0.4567 | 7.734 | 0.00178 | 0.914 |
| ASSB | 13.117 | 0.0206 | 0.5894 | 10.6383 | 0.0013 | 0.979 |
| AASSB | 13.908 | 0.0327 | 0.4858 | 9.478 | 0.0030 | 0.969 |

Table 3: Adsorption isotherms and relevant parameters.

| Adsorbents | Qmax [mg.g ⁻¹] | Langmuir isotherm | | | Freundlich isotherm | | |
|------------|----------------------------|-------------------|-------|----------------|---------------------|--------|----------------|
| | | KL | RL | R ² | n | Kf | R ² |
| ASSP | 224.7 | 0.00197 | 0.911 | 0.9948 | 1.030291 | 0.9996 | 0.9828 |
| ASSB | 384.6 | 0.00242 | 0.892 | 0.9865 | 1.1399 | 1.0443 | 0.9429 |
| AASSB | 400 | 0.00284 | 0.875 | 0.9845 | 1.1941 | 1.1546 | 0.9354 |

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