مجلة جامعة فزان العلمية
Fezzan University scientific Journal
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Adsorption Isotherm of Removal of Heavy Metals in Waste Water by Orange Peels as Low-Cost Adsorbents

*Zamzam Ali Basher Alshreef[1] and Mahjoubah Salih Altayyib Munayr[2]
[1]Department of Analytical chemistry, faculty of Tragen, Fezzan University, Libya
[2] Chemical Engineering Department, faculty of Engineering Sebha University, Libya

Abstract:
The contamination of wastewater by heavy metals represents a critical threat to human health and ecological stability, prompting an urgent search for cost-effective and efficient purification methods. Despite previous advancements, there remains a significant need for research focused on the long-term durability and industrial scalability of various adsorption systems and low-cost sorbents. The objective of this research was to evaluate the efficacy of orange peels as a natural adsorbent for removing heavy metals from wastewater through a series of batch experiments. The adsorption capacity was found to be highly dependent on the contact time. All experimental trials were conducted under a controlled temperature of 30°C.

To characterize the adsorption process, the experimental isotherms were modeled using the Langmuir and the Freundlich equations. Experimental data revealed that the highest removal rates were achieved by orange peels at a contact time of 24 hours. The data indicate that adsorption capacity is highly time-dependent, with optimal removal rates achieved at a 24-hour contact time. Under these conditions, orange peels exhibited the highest removal efficiencies of 99.11%–99.25% for iron, 94.17%–98.76% for copper, and 70.13%–92.30% for nickel. It is worth noting that extending the contact time to 48 hours resulted in increased performance variability and a general decrease in adsorption stability. During this extended period, the removal efficiency decreased to 77.89%–98.09% for iron, 12.98%–99.22% for copper, and 48.45%–95.83% for nickel.

Keywords: Adsorption isotherms, Heavy metals, Orange peels, Wastewater

1. Introduction

The release of toxic heavy metals into the environment poses a severe threat to water quality and aquatic ecosystems, directly endangering human health (Balali-Mood et al., 2021; Türkmen et al., 2022). Trace elements such as arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), mercury (Hg), nickel (Ni), and zinc (Zn) are categorized as primary metallic pollutants in wastewater, industrial effluents, and sewage sludge (Kerur et al., 2021; Qasem et al., 2021; Nejad et al., 2022; Staszak et al., 2023). Traditional methods, including chemical precipitation, membrane filtration, ion exchange, and electroanalysis, are often unsuitable for large-scale industrial applications due to high operational costs and low practical feasibility. Conventional procedures for metal ion removal, such as chemical precipitation, lime coagulation, and reverse osmosis, face limitations like

*Corresponding author: zam.alshreef@fezzanu.edu.ly Received in 26 December2025 – revised form 7 January 2026 Accepted 19 January 2026 - Publication date 24 January 2026

incomplete removal, high reagent demands, and the generation of toxic sludge requiring specialized disposal (Bello et al., 2013).

In contrast, adsorption technology is considered superior for heavy metal removal due to its high efficiency and cost-effectiveness. However, since traditional adsorbents like granular and powdered activated carbon are often economically unviable and technically inefficient (Akesson et al., 2008), recent research has focused on utilizing sustainable, naturally occurring materials and agricultural waste (Dey, 2007; Dey, 2014) as potential sorbents for water purification. Adsorption—the adherence of ions or molecules onto a solid surface—is recognized as an efficient, inexpensive, and eco-friendly technique for treating contaminated water (Pintilie et al., 2007). Complete or partial removal of these toxic metals is essential to prevent health and environmental complications while ensuring ecosystem sustainability (Madhubashani et al., 2021; Chikri et al., 2020). To this end, the World Health Organization (WHO) has established maximum permissible limits for metals like As, Cu, Pb, Cd, Cr, Co, Hg, Ni, and Zn in drinking water. Therefore, it is imperative to develop efficient and environmentally friendly methods to reduce heavy metal content. Among these, adsorption is highly promising due to its simplicity, high efficiency across a wide pH range, and low cost (Anastopoulos et al., 2020). Despite its advantages, the production of commercial adsorbents can be costly, and materials like commercial activated carbons are often non-renewable, making large-scale applications unsustainable (Saravanan et al., 2022). Consequently, it is vital to develop and implement readily available, low-cost, and renewable adsorbents (Fouda-Mbanga et al., 2021). Converting agricultural waste into value-added sorbents aligns with the principles of the circular bio-economy and green chemistry (Etale et al., 2023). Bio-adsorbents offer significant advantages over conventional techniques, providing high affinity and capacity for metal ions (Dey et al., 2021). While biological organisms are limited by their intolerance to low pH or high toxicity (Raji et al., 2023), plant fibers are physically and chemically more robust (Basher et al., 2014). These fibers, composed of cellulose, hemicellulose, lignin, and pectin, bind metal ions through functional groups such as carboxylic, phenolic, hydroxyl, and carbonyl groups. Adsorption can also occur via Van-der-Waals forces or electrostatic interactions at low concentrations (Hartmann et al., 2017). The use of low-cost adsorbents derived from waste—including activated carbon (Padmaja et al., 2017), chitosan, fly ash, or agricultural and industrial residues (Ali et al., 2017)—has gained significant interest. Recent studies highlight the potential of fruit peels for wastewater treatment (Abd-Talib et al., 2020). Specifically, orange peels possess abundant active sites, porous structures, and surface groups that facilitate efficient pollutant adsorption (Liang et al., 2009). The primary objectives of this study were as follows:

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1. Determination of Adsorption Capacity: To quantify the maximum equilibrium uptake of heavy metals by orange peels and evaluate their efficiency under optimized experimental conditions.
2. Adsorption Isotherm Analysis: To analyze the equilibrium data by applying mathematical models, specifically the Langmuir and the Freundlich isotherms, to understand the interaction mechanisms between the adsorbent surface and the metal ions.

2.Experimental

2.1. Preparation of the adsorbent

The raw orange peels were sourced from local waste streams to serve as the precursor material. To ensure the removal of surface contaminants and extraneous matter, the peels were rigorously washed with deionized water. The cleaned biomass was then subjected to air-drying at ambient temperature for a duration of 72 hours until a stable moisture content was achieved. Following the drying period, the material was pulverized using a mechanical laboratory mill to reduce particle size. The orange peels were processed to achieve a uniform particle size of approximately 0.25mm. Finally, the processed powder was stored in hermetically sealed polyethylene containers to maintain its chemical integrity and prevent moisture absorption prior to the adsorption experiments.

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2.2. Collection and Preparation of Wastewater Samples

Wastewater samples were collected from the drainage system in Tragen. Five representative samples were obtained: Sample 1 was collected directly adjacent to the site, while Samples 2, 3, 4, and 5 were taken at distances of 125, 225, 325, and 450 cm, respectively. Immediately following collection, the samples were stabilized via acidification. Concentrated nitric acid (HNO_3) was added until the pH was reduced to below 2 ($pH < 2$). This critical step serves to inhibit microbial activity and maintain heavy metal ions in a stable, soluble state, thereby preventing their adsorption onto the container walls or loss through precipitation. To eliminate the complex organic matrix and ensure all particulate metals were converted into a soluble form, a wet acid digestion protocol was implemented. A precise volume of the preserved sample was transferred to a digestion vessel, to which concentrated HNO_3 was added. The mixture was then heated on a hot plate within a calibrated fume hood. The heating process continued until the sample volume was significantly reduced, reaching a state of near-dryness to facilitate the complete oxidation of organic matter. This cycle of acid addition and heating was repeated until a clear or light-colored, non-carbonaceous solution was obtained, signifying the successful decomposition of the organic matrix. Following the digestion process, the resulting residue was prepared for instrumental analysis. Once cooled to ambient temperature, the residue was filtered using a fine-porosity membrane filter to eliminate any inert suspended solids or particulates that might obstruct the nebulizer or interfere with the Atomic Absorption Spectrometer (AAS) Nova 400 Germany 2003.The filtrate was then quantitatively transferred into a volumetric flask and diluted to a specific mark with ultra-pure deionized water to ensure precise concentration levels. This procedure yielded a stable, homogeneous solution, free of organic interference and optimized for the quantitative determination of heavy metal concentrations via AAS.

2.3. Batch Adsorption Procedure

The adsorption experiments were conducted by introducing 1.0 g of the prepared biosorbent into 15 ml of the aqueous wastewater effluent. These trials were performed at a constant ambient temperature of 30°C for contact periods of 24 and 48 hours to evaluate the effect of time on the adsorption process. Following each specified contact interval, the mixtures were filtered using Whatman No. 4 filter paper to ensure the complete removal of the adsorbent particles. The residual concentrations of heavy metals in the filtrate were subsequently quantified using Atomic Absorption Spectrometry (AAS). The quantity of heavy metals retained by the adsorbent phase (q_e) was determined using the following mass balance equation:

$$q_e = \frac{C_i - C_f}{W} \times V$$

Where:

q_e: Adsorption capacity or the amount of metal adsorbed per unit mass (mg/g), C_i: Initial concentration of the heavy metal in the solution (mg/l), C_f: Final equilibrium concentration of the heavy metal (mg/l), V: Volume of the aqueous solution (l),W: Dry weight of the adsorbent material (g). To ensure the accurate determination of heavy metal concentrations via Atomic Absorption Spectrometry (AAS), the wastewater samples underwent a rigorous preparation process. This procedure was designed to achieve the complete digestion and solubilization of all metallic species within the matrix, thereby ensuring they were in a suitable form for instrumental detection. All experimental trials and subsequent analytical measurements were conducted at the Chemistry Laboratory within the Department of Chemical Engineering, Faculty of Engineering, and the Sebha Central Laboratory.

3.Results and Discussion

3.1. Adsorption Capacity of Heavy Metals

The efficacy of orange peels in sequestering four specific heavy metals was evaluated across five distinct wastewater samples over two contact intervals. As illustrated in Figures 1 and 2, a high initial rate of adsorption was observed, which can be attributed to the abundance of vacant active sites and the large available surface area on the adsorbent during the early stages of the process. The data indicate that the optimal removal efficiency for most heavy metals was achieved at approximately 24 hours of agitation. Beyond this threshold, continued contact resulted in a discernible decrease in the concentration of adsorbed ions, suggesting a potential desorption phase or surface saturation. Notably, as contact time progressed, a general decline in the removal percentages of the four metals was recorded. Comparative analysis of the metallic species revealed that Iron (Fe) exhibited the highest affinity for the orange peel biosorbent at both 24-hour and 48-hour intervals. In contrast, the lowest removal efficiency after 24 hours was observed for Nickel (Ni), while Copper (Cu) showed the minimum adsorption after 48 hours. The results showed that orange peels were remarkably superior in removing iron ions from aqueous solutions compared to other elements. This behavior can be attributed to the combination of some main factors The ionic density and electrostatic attraction. The ferric ion (Fe^{3+}) has the highest charge density due to its high charge and small ionic radius (64 pm). This leads to a superior electrostatic attraction with negatively charged functional groups (such as COO^- groups in pectin) compared to copper (73 pm) and nickel (69 pm) ions. This allows iron to penetrate the micropores of orange peel more easily, which the larger nickel ion might not be able to access. Furthermore, copper (1.90) and nickel (1.91) have a high electronegativity , but iron's higher charge (Fe^{3+}) makes the bonding strength more favorable to iron, which has an electronegativity of 1.83. These findings align with the study conducted by Awash Yirga et al. (2022), which demonstrated the high removal potential of low-cost orange peel powder as a cellulosic adsorbent for Cu(II) and Cd(II) ions in aqueous systems. The comprehensive adsorption capacities for the five wastewater samples after 24 and 48 hours are detailed in Figures 1 and 2, respectively, confirming that orange peels serve as a highly efficient and sustainable medium for heavy metal removal.

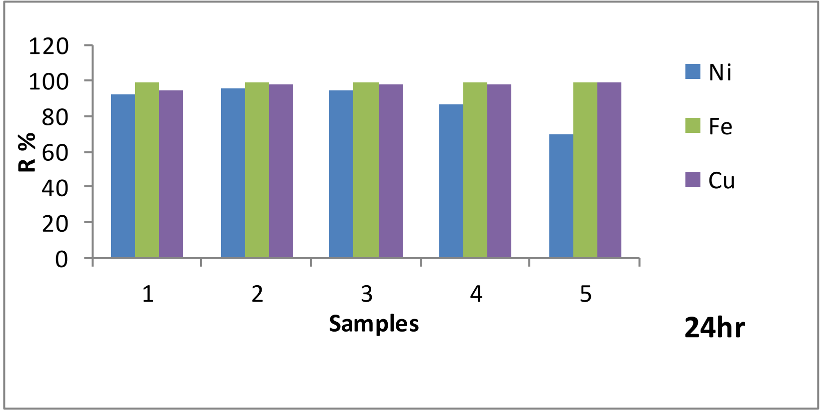

Figure (1): The heavy metals adsorption efficiency for elimination from five wastewater samples via orange peels after 24h contact time.

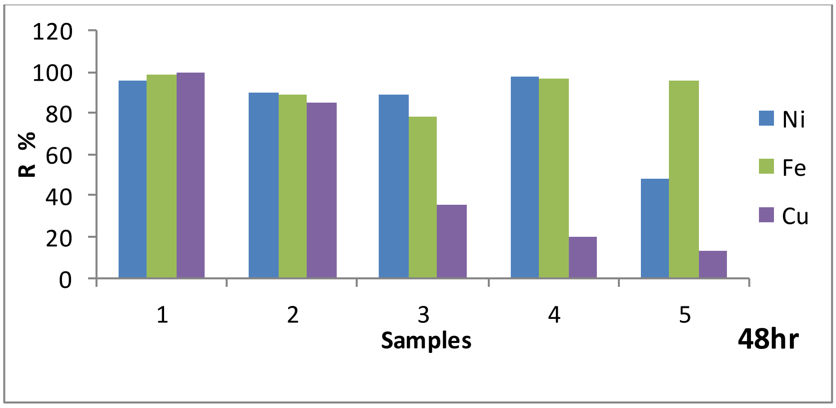

Figure (2): The heavy metals adsorption efficiency for elimination from five wastewater samples via orange peels after 48 h contact time.

The observed decline in adsorption capacity beyond the equilibrium threshold of 24 hours can be attributed to a combination of mechanical and chemical factors. Primarily, desorption phenomena occur due to prolonged mechanical agitation, where kinetic energy disrupts the weak bonds between metal ions and active sites, leading to the re-release of ions into the solution. Furthermore, as the adsorbent surface reaches saturation, electrostatic repulsion between the already-adsorbed ions and those remaining in the aqueous phase hinders additional binding. This is often compounded by the potential structural degradation of the organic matrix in the orange peels, specifically pectin and cellulose, which results in a loss of functional active sites. Additionally, localized pH fluctuations caused by the release of hydrogen protons (H+) during the ion-exchange process increase ionic competition, thereby destabilizing the chemical coordination between the heavy metals and the adsorbent surface.

3.2.Adsorption isotherm

3.2.1. Freundlich adsorption isotherm

The Freundlich model is used to describe the adsorption of solutes from the liquid phase onto a solid surface, and the relationship between the quantity of heavy metals adsorbed per part mass of adsorbent, qe, and the solute concentration at equilibrium, Ce, is shown according to the following equation:

$$q_e\left(mg/g\right) = k\,c_e^{1/n}$$

In this study, qe (represented as x/m) signifies the ratio between the mass of the adsorbed heavy metal (x) and the mass of the orange peel biosorbent (m). As shown in the following plots, the x-axis represents the equilibrium concentration (Ce) of the metal ions in the aqueous phase. The Freundlich isotherm constants for the adsorption of ferric (Fe^{3+}), nickel (Ni^{2+}), and copper (Cu^{2+}) ions onto orange peels were derived from the intercept and slope of the linear plots, as presented below:

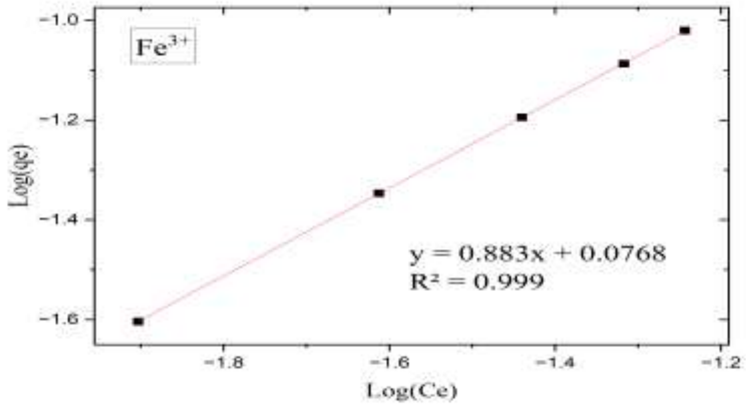

Figure 3: Freundlich adsorption isotherm for Fe^{+3} from wastewater samples onto orange peels

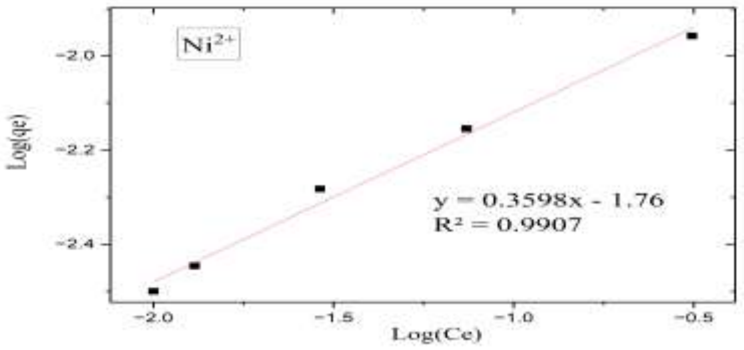

Figure 4: Freundlich adsorption isotherm for Ni^{+2} from wastewater samples onto orange peels

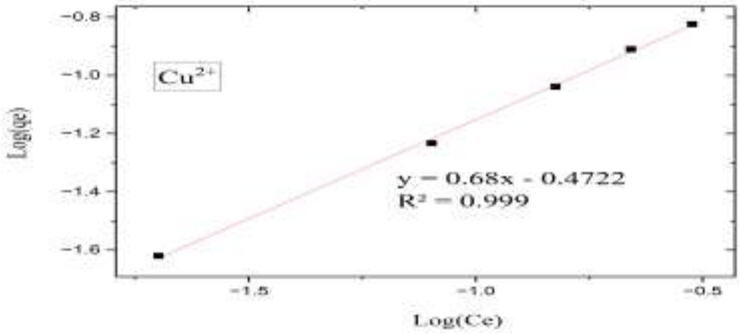

Figure 5: Freundlich adsorption isotherm for Cu^{+2} from wastewater samples onto orange peels

The calculated Freundlich isotherm constants for the adsorption of metal from wastewater samples are summarized in Table 1:

metal	N	K_F	R^2
Fe^{+3}	1.13	1.190	0.9990
Ni^{+2}	2.78	0.017	0.9907
Cu^{+2}	1.47	0.337	0.9990

The adsorption isotherms illustrated in Figures 3–5, along with the numerical parameters summarized in Table 1, demonstrate that the Freundlich model provides an excellent fit for the experimental data. The adsorption intensity constant (n) was determined to be 1.130 for $Fe^{3+,}$ 2.780 for Ni^{2+} and 1.249 for Cu^{2+}. In all instances, the value of n was greater than unity ($n > 1$), which classifies the biosorption process onto orange peels as highly favorable under the tested conditions. Furthermore, the Freundlich capacity constant (K_f) indicates a distinct selective affinity. Based on the calculated values, the adsorption preference of the orange peels follows the order: Fe^{3+} (1.19) > Cu^{2+} (0.337) > Ni^{2+} (0.0173). This hierarchy suggests that the biosorbent has a significantly higher capacity for trivalent iron compared to divalent copper and nickel. The statistical validity of the model was confirmed by the high correlation coefficients (R^2). As presented in Table 1, the R^2 values for all studied metal ions approach unity, indicating that the Freundlich isotherm provides a robust mathematical representation of the adsorption equilibrium. This strong correlation suggests that the adsorption occurs on a heterogeneous surface with a non-uniform distribution of adsorption heat across the biomass.

3.2.2. Langmuir Adsorption Isotherm

The Langmuir isotherm model is based on the fundamental assumption that maximum adsorption occurs when a saturated monolayer of solute molecules forms on the adsorbent surface, which is characterized by a uniform distribution of active sites with constant adsorption energy. In this idealized framework, it is assumed that no lateral interaction or migration of adsorbate molecules occurs between adjacent active sites. Furthermore, the enthalpy of adsorption is considered independent of the degree of surface coverage. The mathematical expression for the Langmuir isotherm is represented by the following equation:

$$\frac{1}{q_e}=\frac{1}{q_{max}.k_L}\times\frac{1}{C_e}+\frac{1}{q_{max}}$$

The essential Langmuir parameters, specifically the maximum monolayer adsorption capacity (q_{max}) and the adsorption energy constant (b or K_L), were derived through the linear regression of the experimental data. By plotting the reciprocal of the equilibrium adsorption capacity ($1/q_e$) against the reciprocal of the equilibrium concentration ($1/C_e$), as illustrated in Figures 6 -8, the constants were accurately determined from the resulting slope and intercept. In this model, q_e denotes the quantity of heavy metal ions sequestered per unit mass of the orange peel adsorbent at equilibrium, while C_e represents the residual concentration of the adsorbate remaining in the aqueous phase. The linearity of these plots serves to validate the applicability of the Langmuir model to the biosorption of the studied metal ions on the homogeneous active sites of the adsorbent.

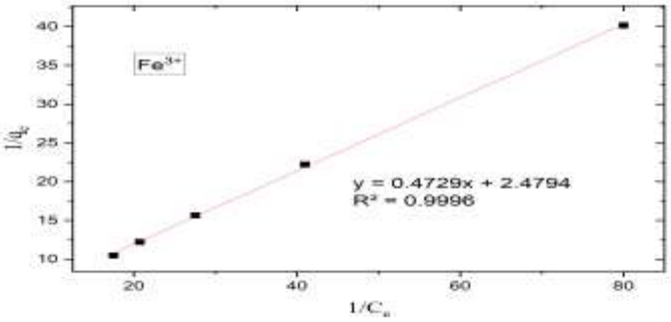

Figure 6. Linearized Langmuir adsorption isotherm for Fe^{+3} onto orange peel in wastewater samples.

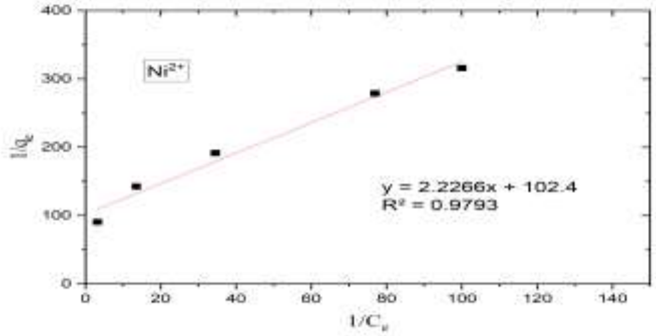

Figure 7. Linearized Langmuir adsorption isotherm for Ni+2onto orange peel in wastewater samples.

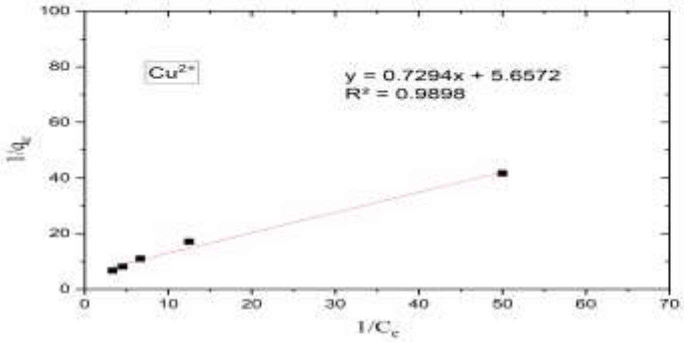

Figure 8. Linearized Langmuir adsorption isotherm for Cu+2 onto orange peel in wastewater samples.

Table 2. Langmuir Isotherm Constants for Metal Ion Adsorption from Wastewater samples .

metal	K_L	q_{max}	R^2
Fe^{+3}	0.1907	2.1146	0.9996
Ni^{+2}	0.0217	0.4491	0.9793
Cu^{+2}	0.1289	1.3710	0.9898

The experimental data were analyzed using both the Langmuir and the Freundlich models to elucidate the adsorption mechanisms of Fe3+, Cu2+, and Ni2+ onto the orange peel biosorbent. The Langmuir constant, qmax, represents the theoretical maximum adsorption capacity required to form a complete monolayer on the adsorbent surface. Quantitative analysis revealed that the orange peels exhibited the highest affinity for Iron (Fe3+), with a qmax of 2.1146 mg/g, suggesting a significantly high density of available active sites for this specific cation. Copper (Cu2+) followed with a capacity of 1.3710 mg/g, while Nickel (Ni2+) demonstrated the lowest capacity at 0.4491 mg/g. These values indicate that the effectiveness of the adsorbent in aqueous purification follows the order: Fe3+ > Cu2+ > Ni2+. The Langmuir affinity constant (KL) provides insight into the bond strength between the metal ions and the adsorbent surface. The KL value for iron (0.19) was considerably higher than that of nickel (0.02), indicating that iron forms more stable and robust chemical bonds with the surface functional groups. These results suggest that the adsorption sites on the orange peels are highly selective toward Fe3+ ions. Statistical evaluation using the correlation coefficient (R2) indicates that the experimental data for all studied metals are in strong agreement with the Langmuir model. For iron, the R2 value of 0.9996 confirms that the process predominantly involves monolayer adsorption on a chemically homogeneous surface where all binding sites possess equivalent energy. Simultaneously, the Freundlich model parameters provided further insight into the nature of the process.

The intensity coefficient (n) for all metal ions ranged from 1.13 to 2.78. Since n > 1 in all cases, the adsorption process is classified as favorable and likely involves physical interactions alongside chemical bonding. Consistent with the Langmuir findings, the Freundlich capacity constant (K_f) was highest for iron (K_f = 1.19), further confirming its superior adsorption potential. In conclusion, while the Langmuir model provides a superior mathematical description for the adsorption of Fe^{3+} and Ni^{2+} based on R^2 values, the adsorption of Cu^{2+} demonstrates excellent compatibility with both the Langmuir and the Freundlich models. This dual compatibility suggests that the copper adsorption mechanism may involve both monolayer formation and interactions on a heterogeneous surface matrix.

4.Conclusion

To evaluate the efficacy of orange peels as a sustainable, low-cost biosorbent for the sequestration of ferric (Fe3+), copper (Cu2+), and nickel (Ni2+) ions from contaminated wastewater. Experimental results identified 24 hours as the optimal contact time for maximum adsorption. During this interval, Iron (Fe3+)exhibited the highest removal efficiency, ranging between 99.11% and 99.25%, while Nickel (Ni2+) removal rates varied from 70.13% to 92.30%. (qmax). According to the Langmuir isotherm model, orange peels demonstrated superior uptake capacity for iron at 2.1146 mg/g, followed by copper at 1.3710 mg/g, and nickel at 0.4491 mg/g. Iron recorded the highest adsorption energy constant (KL = 0.1907), indicating the formation of robust and stable chemical bonds with the surface functional groups, significantly surpassing the affinity observed for nickel (KL = 0.0217). Equilibrium data showed an exceptional fit with the Langmuir model (Correlation coefficient R2 = 0.9996 for iron), confirming a monolayer adsorption mechanism on a chemically homogeneous surface. Additionally, the Freundlich model validated the process as favorable for all studied metals, with intensity parameters (n) exceeding unity (ranging from 1.13 to 2.78). This study underscores that orange peels are highly efficient "bio-adsorbents" characterized by significant selectivity toward iron ions. The findings reveal that extending the contact time to 48 hours leads to a marginal decline in efficiency due to the desorption phenomenon, emphasizing that maintaining the optimal operational time (24 hours) is a critical factor for industrial-scale water treatment applications.

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