



Valorizing Agricultural Waste: Synthesis of Pomegranate Peel-Derived Activated Carbon-Based Hybrid Composite for Sustainable Heavy Metal Removal

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Abstract: The effective removal of heavy metals from water sources is of paramount importance due to their detrimental effects on the environment and human health. In this study, we present a sustainable approach to address this issue by valorizing agricultural waste in the form of pomegranate peel for the synthesis of an activated carbon-based hybrid composite. The pomegranate Peel-Derived activated carbon exhibited high removal capacities for Pb²⁺ and Cd²⁺ ions, with maximum elimination achieved at pH 7 within a pH range of 3 to 11. The adsorption process reached equilibrium in 30 minutes, resulting in remarkable removal efficiencies of 80.3% for Pb²⁺ and 74.3% for Cd²⁺. These findings highlight the successful synthesis and characterization of pomegranate Peel-Derived activated carbon as a promising and environmentally friendly solution for the sustainable removal of heavy metals, emphasizing the significance of agricultural waste valorization in mitigating water contamination.

Keywords: Hybrid Composite, Agricultural waste, pomegranate peel, activated carbon, heavy metal removal, sustainable adsorbents.

Tarımsal Atıkların Değerlendirilmesi: Sürdürülebilir Ağır Metal Giderimi için Nar Kabuğundan Türetilmiş Aktif Karbon Bazlı Hibrit Kompozit Sentezi

Özet: Ağır metallerin su kaynaklarından etkili bir şekilde uzaklaştırılması, çevre ve insan sağlığı üzerindeki zararlı etkileri nedeniyle büyük önem taşımaktadır. Bu çalışmada, aktif karbon bazlı hibrit kompozit sentezi için nar kabuğu şeklindeki tarımsal atıkları değerlendirerek bu sorunu ele almak için sürdürülebilir bir yaklaşım sunuyoruz. Nar kabuğundan türetilen aktif karbon, Pb²⁺ ve Cd²⁺ iyonları için yüksek giderim kapasiteleri sergilemiş ve 3 ila 11 pH aralığında pH 7'de maksimum giderim elde edilmiştir. Adsorpsiyon süreci 30 dakika içinde dengeye ulaşmış ve Pb²⁺ için %80,3 ve Cd²⁺ için %74,3'lük kayda değer giderim verimlilikleri elde edilmiştir. Bu bulgular, nar kabuğundan türetilen aktif karbonun ağır metallerin sürdürülebilir bir şekilde uzaklaştırılması için umut verici ve çevre dostu bir çözüm olarak başarılı bir şekilde sentezlendiğini ve karakterize edildiğini vurgulayarak, su kirliliğinin azaltılmasında tarımsal atıkların değerlendirilmesinin önemini vurgulamaktadır.

Anahtar Kelimeler: Hibrit kompozit, tarımsal atık, nar kabuğu, aktif karbon, ağır metal giderimi, sürdürülebilir adsorbanlar.

1. INTRODUCTION

Since ancient times, heavy metals have been extensively utilized, and their toxicity continues to impact human health. In modern times, the burning of coals abundant with heavy metals in industries has resulted in increased exposure and doses of these metals for individuals [1].

Eco-toxicity, particularly on living organisms, has gained significant attention in recent decades due to rapid urbanization and the continuous pollution of the environment through the discharge of waste and wastewater into ecosystems. Industrial effluents, containing various heavy metal derivatives such as Cd, Pb, Ni, Cr, As, Cu, and Fe, are continuously released, posing a hazardous threat to the aquatic environment [2]. Unlike organic pollutants that can be biologically degraded, metal ions do not decompose into harmless byproducts during degradation [3]. Among the heavy metals, Cadmium (Cd), arsenic, lead, mercury, and chromium lack a biological function and are frequently regarded as a toxicant [4,5].

In recent studies, various agricultural waste materials and biomass-derived sorbents have been investigated for their potential applications in heavy metal ion removal from aqueous solutions. Pomegranate peels, as showcased in the latest research, demonstrated notable promise as a low-cost and eco-friendly biosorbent for the effective removal of Pb^{2+} and Cd^{2+} ions, achieving maximum removal efficiencies at pH 7 within a short contact time. Similarly, Aubergine (eggplant)—based [6] and orange peel-based biosorbents were explored in separate investigations [7], both exhibiting their capacity to adsorb Cu^{2+} , Ni^{2+} , and Co^{2+} ions under specific conditions. These findings underscore the versatility of agricultural waste as sorbent materials, with eggplant stalks additionally explored in a study on activated carbon production. While each study showcases the potential of different biomaterials, the choice of biosorbent, optimal conditions, and adsorption capacities vary, highlighting the importance of tailoring material selection and process optimization for specific heavy metal removal applications. These studies collectively contribute to sustainable solutions for addressing heavy metal pollution in wastewater and offer insights into the utilization of agricultural byproducts in environmental remediation.

Adsorption has emerged as a widely adopted method for removing heavy metals from contaminated water sources. Agricultural waste-derived adsorbents have garnered significant attention due to their high surface area and rich functional groups. Compared to other methods, utilizing agricultural waste for adsorption offers several advantages, including selectivity for specific pollutants, cost-effectiveness, absence of minor hazardous chemicals, and high metal binding effectiveness [5-8]. To meet the requirements of such

activities, activated carbon, with its advantageous physiochemical properties such as excellent mechanical strength, chemical stability in various environments, suitable pore size distribution, and large specific surface area, is considered an essential component [8,9]. Consequently, the synthesis of activated carbons from agricultural by-products has been extensively studied [10].

Numerous agricultural byproducts, such as wood, pine bark, banana pith, cotton and soybean seed hulls, peanut and hazelnut shells, rice husk, wool, orange peel, compost, and others, have been investigated for their potential in metal removal from water [11]. Pomegranate consumption, in the form of fresh fruit, juices, jams, or nutritional supplements, has significantly increased in the past decade due to its associated therapeutic benefits [12,13]. Consequently, substantial quantities of byproducts, particularly peels, and seeds are generated during the industrial processing of pomegranates and are often discarded as waste without being utilized [14].

Pomegranate peel, which accounts for approximately 40 to 50 percent of the fruit's total weight, is particularly rich in phenolic compounds such as flavonoids, phenolic acids, tannins, as well as proteins, bioactive peptides, and polysaccharides [15-17]. In this study, we investigated the sorption of lead and cadmium ions from aqueous solutions using activated carbon produced from pomegranate peel. Specifically, our aim was to develop high surface area activated carbon (AC) from pomegranate peels to effectively remove heavy metals.

2. MATERIALS AND METHOD

2.1. Materials

Sodium hydroxide (NaOH) and cysteine, both sourced from Sigma-Aldrich, were utilized in this study. Cadmium chloride dihydrate ($CdCl_2 \cdot 2H_2O$), hydrochloric acid (HCl), cysteine, sodium hydroxide, and lead nitrate $Pb(NO_3)_2$ were procured from Merck, a reputable chemical supplier. Distilled water was employed to formulate all solutions used in the experimental procedures. Pomegranate peel (PGP) was acquired from a local market situated in Zakho City.

2.2. Method

SEM and EDX analyses were conducted using a Czech Tescan brand model (MIRA III) at the Sharif University of Technology in Tehran, Iran. In contrast, FTIR analysis was performed using a JASCO FTIR-1800 spectrometer at Iraq's Baghdad BPC Analysis Center.

2.1.1. Preparation and characterization of hybrid composite

The extraction of activated carbon from pomegranate peel (PPAC) involved several sequential steps to prepare and activate the raw material. Initially, the PP samples were carefully crushed using a grinder to break them into smaller fragments. To ensure the purity of the peel, the external surface underwent a thorough cleaning process. This involved washing the peel three times with tap water to remove any visible contaminants, followed by three additional washes with distilled water to eliminate any residual impurities. Subsequently, the wet pomegranate peel was allowed to air-dry, ensuring the removal of excess moisture. To further facilitate the drying process and obtain a stable material for experimentation, the peel was subjected to an oven-drying step at a controlled temperature of 105°C for 24 hours. This drying step ensured the complete removal of residual moisture from the peel.

Once the dried pomegranate peel was obtained, it was finely ground into a powder to increase the surface area and facilitate subsequent chemical activation. The resulting powder was stored in an airtight container to maintain its integrity and prevent moisture absorption until further use in the experimental procedures.

A solution comprising cysteine dissolved in sodium hydroxide was prepared for the chemical activation of the pomegranate peel. The pomegranate peel (PP) powder was immersed in this solution and allowed to soak for 5 hours at room temperature. This chemical activation process was designed to enhance pore development within the carbon structure and increase the resulting activated carbon's overall reactivity and adsorption capacity. The presence of cysteine and sodium hydroxide in the activation solution promoted dehydration and oxidation reactions, which facilitated the desired structural and chemical modifications during the synthesis of the hybrid composite (HC).

Subsequently, the activated carbon was obtained through a controlled burning process. The activated pomegranate peel sample was subjected to high temperatures in a muffle furnace, precisely maintained at 650°C by pyrolysis in a closed container for approximately 3 hours, as depicted in Figure 1. At temperatures exceeding 300°C, the volatile components within the carbon matrix began to diffuse out of the pore structure, resulting in the deposition of tarry substances. This step was critical for the removal of these volatile components and the further development of the activated carbon structure.

To ensure the purity of the activated carbon, it underwent multiple washes with distilled water until it reached a neutral pH. This washing step effectively removed any residual chemicals and impurities that may have been generated during the activation process. The activated carbon was then dried in an electric oven

at a temperature of 100°C for 1-2 hours to reduce the moisture content to an optimal level suitable for subsequent experiments.



Figure 1. Schematic diagram of Preparation of hybrid composite.

The activated carbon was further processed by grinding and sieving to obtain uniform particle size, ensuring that particles smaller than 45µm were obtained. These smaller particles have a larger surface area, enhancing their adsorption properties. The activated carbon was carefully stored in a desiccator to maintain its quality by preventing moisture absorption and any potential degradation before its utilization in adsorption experiments. This specific activated carbon derived from pomegranate peel was designated as PPAC [18] representing the 14th batch of activated carbon derived from pomegranate peel.

3. RESULTS AND DISCUSSION

3.1. Fourier Transforms Infrared Spectroscopy (FTIR) For Oxidation Pomegranate Peel Activated Carbon (OPPAC)

The FTIR spectrum of pomegranate peel-activated carbon (OPPAC), as displayed in Figure 2, provides valuable information regarding the surface functional groups present. The presence of these functional groups plays a significant role in determining the properties and potential applications of OPPAC.

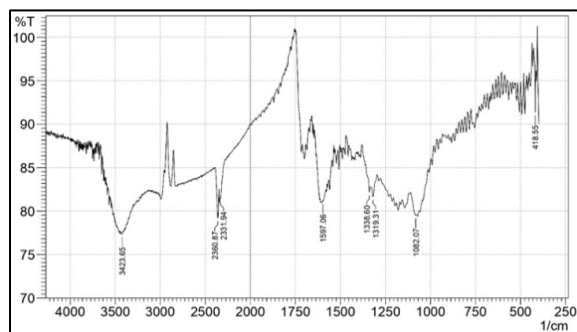


Figure 2. FT-IR spectra of Oxidation Pomegranate Peel Activated Carbon.

One of the prominent bands observed at ν (3441.01) cm^{-1} corresponds to hydroxyl ($-\text{OH}$) groups. This observation suggests the existence of aliphatic alcohol on the surface of OPPAC, indicating the presence of hydrophilic functional groups. Furthermore, a twin peak is observed at ν (1631.78) cm^{-1} and ν (1581.63) cm^{-1} , which can be attributed to the $\text{C}=\text{C}$ stretching vibrations of alkene or conjugated alkene groups. This finding indicates the presence of unsaturated carbon-carbon bonds on the surface of OPPAC, implying the possibility of π -electron conjugation. Additionally, the peak observed at ν (1375.25) cm^{-1} signifies the CH_3 -bending, indicating the presence of methyl groups. This presence of methyl groups suggests the occurrence of aliphatic compounds or substituted aromatic structures on the OPPAC surface. Moreover, the bands observed at ν (1107.14) cm^{-1} and ν (1060.85) cm^{-1} are attributed to the $\text{C}-\text{O}$ stretching vibrations of alcohol groups. This finding suggests the presence of alcohol functional groups, further confirming the existence of hydrophilic moieties on the surface of OPPAC.

Overall, these findings provide valuable insights into the surface chemistry of OPPAC, highlighting the presence of hydroxyl groups, alkene functionalities, methyl groups, and alcohol groups. Understanding these surface functional groups is of paramount importance for comprehending the adsorption behavior and exploring potential applications of OPPAC in various fields [19-22].

3.2. Fourier Transforms Infrared Spectroscopy (FTIR) For Hybrid Composite (HC)

The Fourier Transform Infrared (FTIR) spectrum of the Hybrid Composite (HC) derived from OPPAC is depicted in Figure 3.

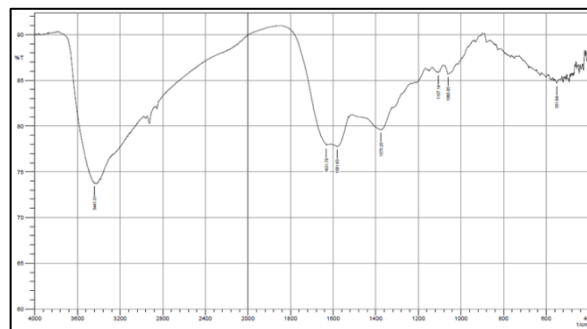


Figure 3. FT-IR spectra of Hybrid Composite (HC) from OPPAC

The prominent peaks observed in the spectrum can be attributed to specific molecular functionalities within the material. Notably, the peak observed at ν (1597.06) cm^{-1} corresponds to the stretching vibration of the nitro compound ($\text{N}-\text{O}$ stretching), indicating the presence of such a functional group in the HC. Furthermore, a twin peak is observed at ν (2360.87) cm^{-1} and ν (2331.94) cm^{-1} , which can be assigned to the stretching vibrations of carbon dioxide ($\text{O}=\text{C}=\text{O}$ stretching) and isocyanate ($\text{N}=\text{C}=\text{O}$ stretching), respectively. These peaks suggest the existence of carbon dioxide and isocyanate moieties within the HC. Another noteworthy peak is observed at ν (1338.60) cm^{-1} , which can be ascribed to the stretching vibrations of $\text{S}=\text{O}$, indicating the presence of sulfur-oxygen bonds. This peak could potentially originate from amino acid compounds, formed because of Cysteine activation. Finally, the peak observed at ν (3423.65) cm^{-1} is attributed to the stretching vibrations of carboxylic acid ($\text{O}-\text{H}$ stretching), suggesting the presence of this functional group in the HC.

Overall, the FTIR spectrum of the HC reveals characteristic peaks corresponding to the nitro compound, carbon dioxide, isocyanate, sulfur oxygen bonds, and carboxylic acid functionalities, providing valuable information about the molecular composition and structure of the material.

3.3. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analysis (EDX) of PP

The surface morphology and elemental composition of polypropylene (PP) are depicted in Figure 4.

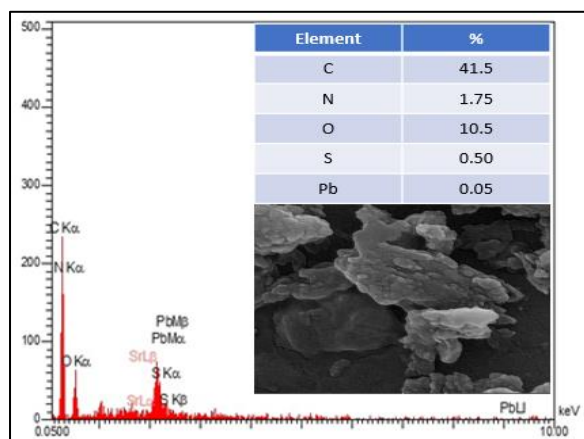


Figure 4. EDX spectrum and SEM photograph of Oxidation Pomegranate Peel Activated Carbon

The analysis revealed that PP consists of various chemical elements, with carbon (C) comprising approximately 41.5% of the composition. Nitrogen (N) constitutes around 1.75%, oxygen (O) accounts for approximately 10.5%, and sulfur (S) is present at a concentration of approximately 0.50%. Furthermore, a small amount of lead (Pb), representing about 0.05% of the composition, was detected in the PP sample after Pb^{2+} was adsorbed from wastewater to detect the amount (Pb).

These findings provide valuable insights into the elemental composition of PP, allowing for a better understanding of its chemical makeup and potential implications for its properties and applications. The surface morphology and chemical ingredients of PP are given in Figure 4.

3.4. EDX of removal Cd by Hybrid material

The EDX spectrum and SEM photograph of the Oxidation of Pomegranate Peel Activated Carbon after Cd^{2+} ion adsorption are presented in Figure 5.

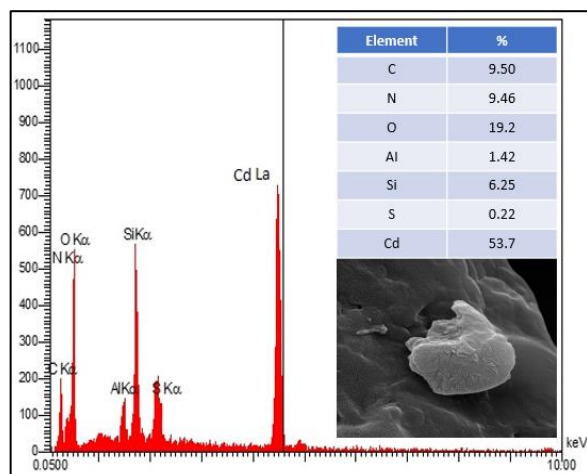


Figure 5. EDX spectrum and SEM photograph HC after Cd^{2+} ion adsorption

The EDX spectrum and SEM image of the specimen provided compelling evidence for the presence of carbon (C), nitrogen (N), oxygen (O), sulfur (S), and cadmium (Cd) atoms. The chemical composition analysis further confirmed the incorporation of these elements into the hybrid material following the adsorption of Cd^{2+} ions from the solution. Based on the EDX spectrum, the combined adsorbents demonstrated the following elemental composition: 9.50% C, 9.46% N, 19.2% O, 1.42% Al, 6.25% Si, 0.22% S, and 53.7% Cd^{2+} ions. These results highlight the significant presence of carbon, nitrogen, oxygen, and cadmium within the hybrid composite after the adsorption process. The comprehensive characterization through EDX analysis, SEM imaging, and chemical composition analysis provides valuable insights into the elemental composition and the successful incorporation of Cd^{2+} ions into the hybrid material.

3.5. Adsorption Studies

3.5.1. Effect of pH

The pH of the solution plays a crucial role in the adsorption process of metals from aqueous solutions, primarily due to its impact on the surface charge of the adsorbent [21]. To investigate the effect of pH on the HC, a series of experiments were conducted by adjusting the pH of the solution using 0.1 M HCl and 0.1 M NaOH as buffer solutions. The pH range studied varied from 3 to 13, with all experiments conducted at room temperature (27°C) and using a fixed initial concentration of HAC (100 mg/L) with an agitation speed of 125 rpm (Fig. 6).

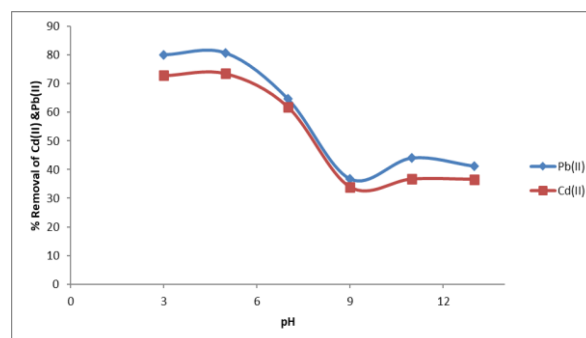


Figure 6. The Effect of pH on Adsorption.

As shown in Figure 5.1, the results indicate that the maximum removal percentages of the Pb^{2+} and Cd^{2+} ions from the adsorbents were achieved at pH levels between 3 and 6, with removal efficiencies of 80.7% and 73.5%, respectively. However, as the pH values increased beyond this range, the removal percentages dramatically decreased. Notably, there was only a slight increase in removal efficiency after reaching a pH of 9. These findings demonstrate the pH-dependent

nature of the adsorption process, with optimal removal occurring at acidic to slightly acidic conditions.

The variation in surface charge of the HC at different pH levels is likely responsible for the observed changes in metal ion adsorption efficiency. The pH dependency should be considered when designing adsorption processes for the removal of the Pb^{2+} and Cd^{2+} ions using Hybrid Activated Carbon materials.

3.5.2. Effect of Time

The effect of contact time on the removal of lead (Pb^{2+}) and cadmium (Cd^{2+}) using various adsorbent materials was investigated in this study. The contact times were varied from 10 minutes to 70 minutes, as shown in Figure 3.6. The aim was to determine the time required for the adsorption process to reach equilibrium. Each adsorbent material was tested with an initial solution pH ranging from 6.7 to 7, using 0.3 grams of the adsorbent.

The results, depicted in Figure 7, revealed a rapid initial uptake rate of Pb^{2+} and Cd^{2+} adsorption within the first 30 minutes. After this period, the adsorption rate became constant. It was observed that equilibrium was reached after 70 minutes, with final adsorption percentages of 80.3% for Pb^{2+} and 74.3% for Cd^{2+} .

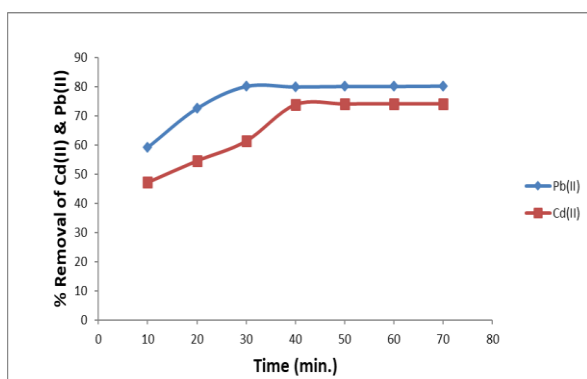


Figure 7. The Effect of Time on Adsorption

The initial adsorption rate is influenced by the availability of unoccupied sites on the surface of the carbon-based adsorbents. Consequently, the adsorption of Pb^{2+} and Cd^{2+} ions was relatively high during this period. As the contact time increased, the extent of adsorption for Pb^{2+} and Cd^{2+} ions remained constant until the process reached equilibrium. This adsorption behavior can be attributed to external surface diffusion.

In summary, the findings of this study demonstrate that increasing the contact time between the adsorbent materials and the Pb^{2+} and Cd^{2+} ions enhances their removal efficiency. The initial rapid uptake rate followed by a constant adsorption rate indicates the importance of allowing sufficient contact time for the effective adsorption of these heavy metal species.

3.5.3. Effect of Initial Metal Ion Concentration

The mechanism of metal uptake is highly influenced by the initial concentration of heavy metals. At low concentrations, metals are adsorbed by specific sites on the adsorbent material. However, as the metal concentration increases, these specific sites become saturated, and the adsorbent surface exchange sites become occupied [22].

Figure 8 illustrates the impact of metal concentration on the percentage removal of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions. The results demonstrate that as the initial concentrations of these metals increase, the percentage removal decreases. Additionally, the figure highlights that HC, among all the types of adsorbent materials used, exhibits the highest percentage of removal. The observed decrease in percentage removal with increasing initial metal concentrations can be attributed to several factors.

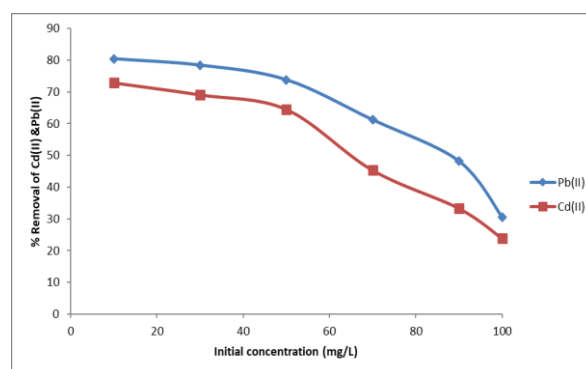


Figure 8. Effect of initial metal ion concentration

At higher metal concentrations, there is greater competition for available adsorption sites, resulting in reduced adsorption efficiency. The limited number of specific sites and exchange sites become overwhelmed by the high metal concentrations, leading to a decrease in the overall removal percentage.

4. CONCLUSIONS

The presence of heavy metal ions in wastewater poses significant concerns for both ecological and human health safety. In this study, we developed and investigated the effectiveness of a novel, low-cost, and eco-friendly biosorbent derived from pomegranate peels for the removal of Pb^{2+} and Cd^{2+} heavy metal ions from wastewater. The experimental results demonstrated that the synthesized pomegranate peel biosorbents exhibited high removal capacities for Pb^{2+} and Cd^{2+} ions, with maximum elimination achieved at pH 7 within a pH range of 3 to 11. The adsorption process reached equilibrium at a contact time of 30 minutes, resulting in a remarkable removal efficiency of 80.3% for Pb^{2+} and 74.3% for Cd^{2+} . Furthermore, the impact of initial metal ion concentration was

investigated, revealing a noteworthy trend. As the initial concentrations of the metal ions increased, the percentage of metal removal decreased. This finding suggests that at higher metal concentrations, the adsorption sites on the pomegranate peel biosorbents become overwhelmed, leading to a reduced removal capacity. In conclusion, this study successfully demonstrated the potential of utilizing pomegranate peel-based biosorbents as a viable and sustainable solution for the removal of Pb^{2+} and Cd^{2+} heavy metal ions from wastewater. The optimized conditions, such as pH 7 and a contact time of 30 minutes, exhibited the highest removal efficiency. However, conducting further research to optimize the biosorbent synthesis and explore its performance under different operating conditions is important. Additionally, the potential of scaling up this biosorption process for practical applications in wastewater treatment should be investigated.

Conflicts of Interest: The authors declare no competing financial interests.

Ethical Approval: Ethical approval is not required for this study.

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