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ORIGINAL ARTICLE

The Removing Cadmium Ions from Aqueous Solutions Using Barley Straw: An Experimental Study

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ABSTRACT: Cadmium is a toxic metal that can contaminate water sources and pose serious health risks to humans and the environment. Therefore, there is a need for developing low-cost and eco-friendly methods for cadmium removal from water. In this research, we investigated the efficacy of barley straw in removing cadmium ions from aqueous solutions. The adsorbent utilized was laboratory-scaled barley straw that underwent pulverization via standard ASTM sieves, specifically those within the 40 to 120-mesh sieve size range. The functionalization of barley straw was achieved via treatment with a 0.8 M NaOH solution. The adsorbent was subsequently characterized by FTIR analysis to identify the presence of functional groups. The FTIR analysis indicated that the modification of barley straw led to an elevation in the stretch vibration band of hydroxyl and carboxyl groups. At the optimized experimental condition, a cadmium removal efficiency of up to 98.60% was achieved. These results demonstrate the potential of barley straw as an effective adsorbent for removing cadmium ions from aqueous solutions.

INTRODUCTION

Heavy metals are a group of elements with high atomic weights and densities, including lead (Pb), cadmium (Cd), mercury (Hg), and arsenic (As) [1,2]. Although some of these elements are essential for human health in small amounts, they can be toxic in larger amounts [3]. They can enter the environment through natural processes such as volcanic activity and erosion, as well as through human activities such as industrial processes, mining, and the use of certain pesticides and fertilizers [2]. Cadmium is a soft, bluish-white metal that is commonly found in the Earth's crust. It has a boiling point of 767°C and a melting point of 321°C [4]. It is often used in various industrial applications, including batteries, pigments, coatings, and alloys. However, it is also a toxic heavy metal and constitutes a serious threat to human health and the environment. Exposure to

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cadmium can cause various health problems, including kidney damage, lung damage, and cancer [5,6].

The main sources of cadmium exposure for humans are contaminated air, water, and food. Cadmium can be released into the environment from industrial activities such as mining, smelting, and refining [7]. In addition, cigarette smoke is a significant source of cadmium exposure, with one cigarette containing up to 1.7 µg of cadmium [8].

The World Health Organization (WHO) has established a provisional tolerable monthly intake (PTMI) of 25 µg of cadmium per kilogram of body weight [9]. Workers in various industries, including mining, battery manufacturing, and welding, are at high risk of cadmium exposure. According to the Occupational Safety and Health Administration (OSHA), the permissible exposure limit (PEL) for cadmium in the workplace is 5 µg $m^{3-1}$of air as an 8-hour time-weighted average [10]. The cadmium in drinking water has a maximum contaminant level (MCL) of 5 g L^{-1}which has been imposed by the United States Environmental Protection Agency (EPA) [11].

Therefore, it is imperative to remove heavy metal ions from freshwater and contaminated water. Several methods can be employed to remove heavy metal ions from contaminated water including chemical precipitation, adsorption, ion exchange, and membrane filtration [12–16]. Chemical precipitation involves adding chemicals like lime, sodium hydroxide, and sodium carbonate to contaminated water, which then precipitate heavy metal ions as insoluble compounds. However, this method requires careful pH control and the proper disposal of resulting sludge. Adsorption, a widely used method, employs adsorbents such as activated carbon, zeolites, and clay minerals with high affinity for heavy metal ions. The adsorbents can be in the form of a powder, granules, or beads, making it simple, cost-effective, and producing little waste. Ion exchange is another method that involves exchanging heavy metal ions with ions of opposite charge immobilized on a resin. This resin, which can be in the form of beads or a column, is charged with an ion that has the same charge as the heavy metal ions to be removed. While effective, ion exchange requires expensive resins and careful management of the resin bed. Membrane filtration, which utilizes membranes such as reverse osmosis and nanofiltration, is also an effective method that produces little waste and can treat large volumes of water. These membranes have pores that are small enough to allow the passage of water molecules but prevent the passage of heavy metal ions.

Surface adsorption is currently one of the most widely used methods for purifying heavy metals from contaminated water [17]. The cornerstone of this technology is the adsorbent material, which has been utilized in a range of forms to eliminate heavy metals from water and wastewater. In real-world scenarios, the utilization of typical adsorbents is linked with difficulties related to their restricted adsorption capacity, sluggish adsorption rate, or inadequate regeneration capabilities [18,19]. In recent years, the utilization of agricultural waste as an adsorbent in surface adsorption processes has attracted the attention of numerous researchers due to its cost-effectiveness [20–22].

During a meeting held by the Ion Exchange Group of Chemical Industries and Extractive Engineering in England in the early part of 1986, bio-adsorbent was presented as a novel technology [23]. Since then, there has been a surge of interest worldwide in the area of bio-adsorption, with the aim of identifying possible bio-adsorbents. Previous studies have shown that bio-adsorption is a highly effective approach for the treatment of wastewater that contains metals [24]. Since the late 1990s, there has been a shift in the approach to adsorption studies using plant residues and agricultural waste [25]. Rather than relying on physical activation methods such as activated carbon, there has been a move towards utilizing a chemical improvement process. This method is more cost-effective than physical activation, while still increasing adsorption capacity.

The aim of this research was to explore the capability of using barley straw plant residues as an affordable and readily available adsorbent for cadmium ion removal from an aqueous solution. Additionally, this research aimed to enhance the adsorption capacity of barley straw by treating it with alkaline NaOH solution. The impact of various factors, including the adsorbent size (mesh sieve), contact duration, pH of the solution, and adsorbent dose were investigated to determine their effect on the rate of cadmium adsorption.

MATERIAL AND METHODS

The barley straw utilized in this research was sourced from farms located in Al-Qadisiyah governorate, Iraq. To prepare the barley straw waste adsorbent for use, it underwent a thorough washing process using deionized water to remove any dust present. Following this, the barley straw was dried at 60°C for 12 hours in a thermal oven and subsequently ground into powder. The powder was then sieved through a mesh with a range of 40 to 120 using a sieve shaker. To improve the adsorbent material, 100 g of normal barley straw with the optimal size was added to a 2 L solution of 0.8 M NaOH. The mixture was then stirred for a duration of 5 hours. Following this, the resulting samples with varying sizes were placed in sealed plastic bags. After the activation period, the improved barley straw was thoroughly rinsed with distilled water to eliminate any remaining excess NaOH. The resulting improved adsorbents were then dried in an oven at 90°C for a duration of 6 hours, and later stored in individual containers [26].

The type of materials used along with the specifications were cadmium nitrate ($Cd(NO_3)_2.4H_2O$), hydrochloric acid (HCl), sodium hydroxide (NaOH), and nitric acid (HNO_3) which were used as removal agent, pH adjusting agent, an agent for adjusting and improving pH, and dispersant agent, respectively. Merck, a science and technology company headquartered in Darmstadt, Germany, provided the materials.

To identify the samples and determine the functional groups and bonds in their molecules, Fourier-transform infrared (FTIR) spectroscopy was employed (Nicolet 6700; Thermo Fisher Scientific Inc., Waltham, MA, USA). The samples were added separately to potassium bromide (KBr) salt and thoroughly ground in an agate mortar. The resulting mixture was then compressed into tablets using pressure to create a uniform matrix. These tablets were loaded into a holder and subjected to FTIR spectrophotometry to obtain spectra in the 400 to 4000 cm^{-1} wavelength range. This allowed for a qualitative analysis of the samples.

An atomic absorption spectrometer (PerkinElmer SIMAA 6000), along with an air acetylene flame and a hollow cathode lamp tailored for detecting cadmium, were employed to measure the concentration of cadmium before and after equilibrium. To generate a calibration curve within the linear range for the cadmium cation, the initial step involved preparing a cadmium stock solution with a concentration of 1 g L^{-1}. To prevent the formation of cadmium hydroxide ($Cd(OH)_2$) precipitate, 2.5 mL of HNO_3 was added to a 10 mg L^{-1}solution that was created after successive dilutions.

To conduct the experiments, adsorbent was added to multiple polyethylene containers with lids, each containing 100 mL of a 20 mg L^{-1} cadmium-concentrated solution. The required amount of adsorbent was added first. The resulting samples were mixed at ambient temperature with a mixing speed of 250 rpm. Upon completion of the equilibrium time of 20 min, the samples were taken off the shaker and using filter paper, the adsorbent was separated from the solution. The atomic absorption device was used to measure the concentration of cadmium solutions that were not affected by adsorbent. Subsequently, the equation $\mathrm{q_e = (C_i - C_e)V/M}$ was employed to determine the quantity of cadmium that had been absorbed by the adsorbent [27,28]. In this equation, $\mathrm{q_e}$ is the amount of ion absorbed by the adsorbent at equilibrium (mg g^{-1}), and $\mathrm{C_e}$ and $\mathrm{C_i}$ are the equilibrium (final) and initial concentrations of cadmium cations in the solution (mg L^{-1}). M is the mass of adsorbent in g, and V is the solution volume in L. The value of the percentage of cadmium ion adsorption by the adsorbent was calculated using $100(C_i - C_e)/C_i$ [29–31].

In the present study, the adsorption process involves four control variables, and altering each of them can impact the cadmium removal efficiency. Table 1 outlines the investigated factors and experimental conditions for the absorption of cadmium using a modified barley straw adsorbent.

Table 1. The variables considered in studying the impact of cadmium removal efficiency.

Parameters	Range	Experimental conditions
Adsorbent size	40-120	Contact duration: 30 min, initial concentration of cadmium: 20 mg L^{-1}, adsorbent dose: 0.1 g, pH: 5, stirring speed: 250 rpm
pH	2-8	Contact duration: 20 min, adsorbent dose: 0.1 g, initial concentration of cadmium: 20 mg L^{-1}, adsorbent size: 80-mesh sieve, stirring speed: 250 rpm
Contact duration (min)	5-45	Adsorbent dose: 0.1 g, Initial concentration of cadmium: 20 mg L^{-1}, adsorbent size: 80-mesh sieve, pH: 5, stirring speed: 250 rpm
Adsorbent dose (g)	0.05-3	Contact duration: 20 min, initial concentration of cadmium: 20 mg L^{-1}, pH: 6, adsorbent size: 80-mesh sieve, stirring speed: 250 rpm

RESULTS AND DISCUSSION

Infrared spectroscopy is a widely used analytical technique in chemistry that allows the identification of functional groups and the characterization of molecular structures. It works by measuring the interaction of infrared radiation with the chemical bonds in a sample. Figure 1 displays the infrared spectrum of the normal and improved barley straw when in contact with a solution. The peak observed at 3400 cm^{-1} corresponds to the O-H vibrations associated with barley straw, indicating the existence of hydroxyl group on its surface. This spectral band corresponds to the oscillations of the silanol (≡Si-OH) group, which is attached to the hydroxyl group in cellulose and lignin, and water molecules adsorbed on the surface of barley straw. The peaks identified at 2920 cm^{-1} correspond to the stretching vibrations of the C-H band present in the methyl group. The peaks detected at 1730 cm^{-1} indicate the stretching vibrations of the carbonyl group specific to ketones. The peaks found in the range of 1510 cm^{-1} can be associated to the stretching vibrations of the C=C bond in the aromatic ring. The peaks around 1200 cm^{-1} could correspond to the stretching vibrations of the C-O group in phenolic compounds.

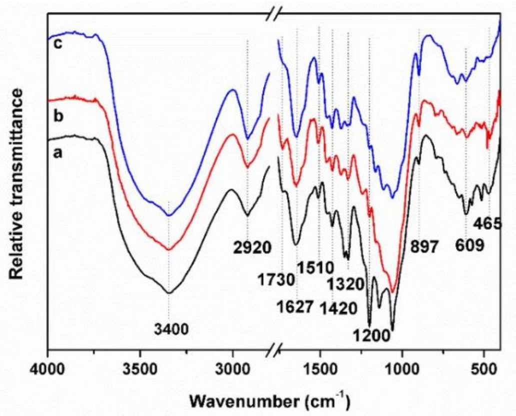

Figure 1. Fourier Transform Infrared (FTIR) spectra of three different samples: (a) barley straw in its normal state, (b) improved barley straw, and (c) improved barley straw after exposure to a cadmium solution.

The improvement of the adsorbent resulted in a significant strengthening of the O-H vibration, as indicated by the shift of the 3335 cm^{-1} peak to 3400 cm^{-1}. Furthermore, the shift of the O-H vibration peak at 3400 cm^{-1} to 3390 cm^{-1} reflects the impact of the improved adsorbent on the reaction with cadmium ions. During the adsorption process, the presence of cadmium ions in the solution caused the peak at 2920 cm^{-1} to shift to 2900 cm^{-1}, indicating their involvement in the process.

To analyze adsorbent size, barley straw samples weighing 0.1 g each were subjected to testing with 20 ppm cadmium solution for 30 min. The samples were divided into various sizes ranging from 40 to 120-mesh sieve. According to Table 2, the cadmium removal efficiency of these particles increases with a decrease in particle size, which is primarily due to the significant increase in surface area for mass transfer.

Table 2. Cadmium removal efficiency vs. the adsorbent size (mesh sieve). The initial cadmium concentration was 20 mg L^{-1}, the pH was 5, the adsorbent dose was 0.1 g, and the contact duration was 30 min.

Mesh sieve size	**40**	**50**	**60**	**70**	**80**	**90**	**100**	**110**	**120**
Cadmium removal efficiency, %	58.85	64.20	68.48	74.90	92.02	92.02	92.02	92.02	92.02

Smaller particles tend to be absorbed more quickly than larger particles, increasing the likelihood of adsorption on the outer surface of the adsorbent particles. By decreasing the particle size, there is also a reduced probability of metal ions penetrating from the external surface into the internal cavities of the absorber. However, smaller particles have lower penetration resistance for mass transfer. Various factors can negatively impact cadmium removal efficiency in larger particle sizes, including the length of the permeation path, mass transfer resistance, and blockage of certain permeation paths, resulting in underutilization of the internal surfaces of the particles during adsorption. To maximize cadmium removal efficiency, it was observed that increasing the effective surface up to 80 mesh size was beneficial, with percentage removal of cadmium remaining consistent even after increasing the mesh up to 120. Therefore, experiments in subsequent stages were conducted using an adsorbent size of 80-mesh sieve.

To study the impact of contact duration and identify the point of equilibrium, we exposed 0.1 g of normal and improved barley straw to 100 mL of 20 ppm cadmium solution for varying durations. According to Table 3, in laboratory conditions the normal barley straw required 30 min to reach equilibrium for cadmium removal, while the improved barley straw only needed 20 minutes.

Table 3. Cadmium removal efficiency vs. contact duration. The initial cadmium concentration was 20 mg L^{-1}, the pH was 5, and the adsorbent dose was 0.1 g.

Contact duration (min)	**5**	**10**	**15**	**20**	**25**	**30**	**35**	**40**	**45**
Normal barley straw	36.75	56.70	67.20	77.70	86.21	86.21	86.21	86.21	86.21
Improved barley straw	52.50	75.00	85.16	97.62	97.62	97.62	97.62	97.62	97.62

Once equilibrium was reached, the normal barley straw achieved a removal percentage of 87.6%, while the improved barley straw had a higher cadmium removal percentage of 96.4%. The experiments revealed that in the initial stages, the concentration of cadmium ions was high, leading to more effective collisions with the surface sites on the barley straw. This resulted in a higher removal percentage at a faster rate. However, as time passed, the removal of cadmium cations decreased due to saturation of the surface sites on the barley straw. Consequently, the removal rate also decreased.

The pH level is a crucial environmental factor in cadmium removal as it not only impacts the absorption of cadmium cations but also leads to hydrolysis and the formation of hydroxide precipitates. To achieve this goal, the pH PZC value of the enhanced barley straw adsorbent was established, followed by an examination of how the pH level influences the extent of cadmium removal via the improved barley straw adsorbent. To ascertain the pH PZC value of the adsorbent under investigation, 10 samples of 50 mL 0.01 M potassium nitrate (KNO_3) solution were prepared and their initial pH levels were adjusted using HNO_3 and NaOH. Next, a specific quantity of altered adsorbent was introduced into each sample and agitated for six hours. The resulting pH of the solution was subsequently determined. Based on the findings, the pH PZC of the improved barley straw adsorbent was determined to be 8.4 (Figure 2).

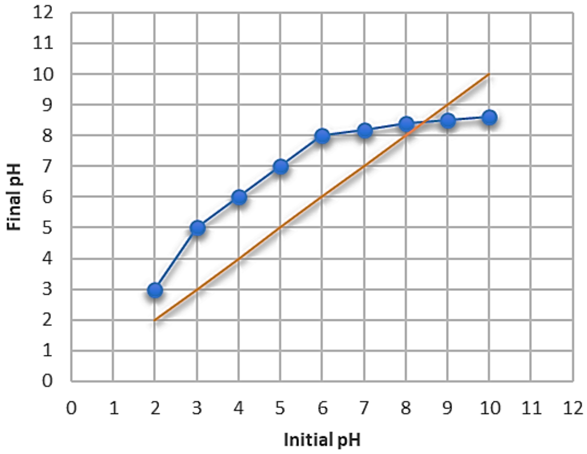

Figure 2. Determination of pH PZC for sorbent.

In pH PZC, the absorbent surface becomes neutral in charge, which makes it easier for cations to be absorbed. However, increasing the pH above pH PZC and increasing the concentration of hydroxide ions results in a negative charge on the surface of the absorbent, which increases the attraction between the absorbent and cations, leading to greater cation removal.

Table 4 presents the outcomes of the investigation on how pH affects the absorption of cadmium metal. The study was conducted under specific test conditions, including a pH range of 2 to 8, an adsorbent dose of 0.1 g, an initial concentration of 20 mg L^{-1}, a contact duration of 20 min, and a mixing speed of 250 rpm, for the improved barley straw adsorbent. Additionally, at a temperature of 30±1°C the experiments were carried out.

Table 4. Cadmium removal efficiency vs. pH. The initial cadmium concentration was 20 mg L^{-1}, the adsorbent dose was 0.1 g, and the contact duration was 20 min.

pH	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Cadmium removal efficiency (%)	45.89	66.74	82.40	90.64	97.85	82.40	61.80

It is evident that as the pH increases from 2 to 6, there is an increase in absorption. The alteration of pH impacts the level of absorption by modifying the ionization level of the adsorbed species. Since M^{2+} divalent metal ions can take various forms in water solutions, the M^{2+} species that is most appropriate for absorption on the desired absorbent surface is the dominant species for each cation at a specific pH. When the pH is high, metal hydroxide formation occurs alongside the absorption process, effectively removing metal species from the solution. However, when the pH is low, the presence of H_3O^+ and M^{2+} competing against each other reduces the capacity for metal cation adsorption. Table 4 shows that the rate of cadmium absorption rises as the pH increases. Therefore, in the continuation of the research, the optimal pH for cadmium absorption on improved barley straw was selected as 6. In studies conducted by other researchers, it has been shown that the optimum pH for cadmium removal is in this range [27–29].

To explore the impact of the quantity of adsorbent on the adsorption of cadmium, tests were conducted on a solution containing 20 mg L^{-1} of cadmium. The adsorbent was varied from 0.05 to 3 g, and at a temperature of 30°C the experiments were carried out. Based on the results of experiments, it has been determined that the ideal quantity of absorbent for enhancing barley straw is 1.0 g. The findings suggest that the adsorption capacity and efficiency in removing cadmium metal increase with an increase in the amount of adsorbent due to the availability of more adsorption sites. Therefore, it can be concluded that a higher quantity of absorbent leads to better absorption performance. The rise in the absorption rate can be attributed to the greater amount of adsorbent utilized. Table 5 shows how the quantity of cadmium cations absorbed is impacted by the amount of adsorbent used.

Table 5. Cadmium removal efficiency vs. adsorbent dose. The initial cadmium concentration was 20 mg L^{-1}, the pH was 6, and the contact duration was 20 min.

Adsorbent dose (g)	**0.05**	**0.1**	**0.5**	**1**	**1.5**	**2**	**2.5**	**3**
Cadmium removal efficiency, %	85.85	90.90	97.97	98.60	98.60	98.60	98.60	98.60

In relation to the impact of the quantity of adsorbent on the amount of cadmium absorbed, it can be inferred that as the quantity of adsorbent increases, the proportion of each adsorbent type decreases relative to the total amount of adsorbent, resulting in a reduction in the amount of cadmium absorbed. In this regard, a study used banana peel adsorbent improved with sodium hydroxide to remove cadmium metal ions. The obtained results showed that banana peel improved with 0.8 M sodium hydroxide solution has a higher absorption capacity than banana peel and 93.4% cadmium removal efficiency was achieved [32].

CONCLUSIONS

The purpose of this research was to investigate the feasibility of removing cadmium from aqueous solutions using improved barley straw as an adsorbent. Cadmium is a toxic heavy metal that can cause serious health problems, and its presence in the environment is a major concern. The researchers conducted the study using a batch process in the laboratory. They prepared aqueous solutions containing a known concentration of cadmium and added the improved barley straw as an adsorbent. The system was allowed to interact for a predetermined period, after which the concentration of cadmium in the solution was measured to determine the cadmium removal efficiency. The findings of the study were promising. The researchers found that the improved barley straw was a cost-effective and highly effective adsorbent under optimal laboratory conditions. Specifically, the discontinuous system yielded a 98.6% cadmium removal rate within just 20 minutes of contact time using the improved barley straw adsorbent. These results suggest that improved barley straw could be a promising solution for removing cadmium from contaminated water sources. The study provides a foundation for further research to explore the use of this adsorbent in larger-scale applications and under different environmental conditions. Overall, this study demonstrates the potential of using sustainable and low-cost agricultural waste materials as effective adsorbents for environmental remediation.

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Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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