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Article

From agricultural waste to water purifier: Walnut Shell activated carbon for cadmium ion adsorption

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Abstract: This work investigates the use of activated carbon in a batch system for the adsorption of cadmium from aqueous solutions. Two adsorbents were assessed: Commercially available activated carbon (CAC) and synthesized activated carbon (SAC) produced from walnut shell–derived agricultural waste. Adsorption efficiency increased with increasing pH, according to research on the impact of solution pH on cadmium removal. In the pH range of 7–12, maximum removal efficiencies of 81.9% for SAC and 80.5% for CAC were found at an initial cadmium concentration of 100 mg/L. Freundlich and Langmuir isotherm models were used to analyze the adsorption data. The Freundlich model provided a marginally better fit, suggesting heterogeneous adsorption sites on the activated carbon surfaces, while both models demonstrated good agreement with the experimental results. The highest adsorption capacities that were obtained from adsorption isotherms, under conditions of 4 h of contact time and 30 °C, were 69.5 mg/g for SAC and 81.2 mg/g for CAC.

Keywords: cadmium ion adsorption; walnut shell; activated carbon; Freundlich isotherm; Langmuir isotherm; water purification

1. Introduction

Heavy metal pollution of water, particularly cadmium (Cd^{2+}), is a serious environmental and human health hazard [1,2]. Cadmium is a persistent, non-biodegradable heavy metal that accumulates in aquatic environments and can be transferred through the food chain to humans, where it is associated with severe adverse health effects, including kidney damage, skeletal demineralization, and carcinogenesis[3]. Electroplating, mining, and battery manufacturing are the main causes of cadmium contamination in the water bodies [4]. The World Health Organization (WHO) and other international standards provide the maximum concentration of cadmium in drinking water as 0.003 mg/L (3 µg/L) to ensure the population is not at risk of health complications. Most industrial effluents, however, go beyond this limit and create a problem in the treatment of water sources and become a burden to water treatment plants [5]. Different processes have been established to eliminate the presence of heavy metals in contaminated water and solutions, such as chemical precipitation, solvent extraction, electrolytic recovery, oxidation, surface adsorption, osmosis, molecular ion exchange, evaporation, and dilution as well as filtration and flotation [6]. All these approaches have particular strengths and weaknesses and tend to strike a balance between price, efficiency, and complexity. Adsorption has become one of the most promising methods because of its high purifying capacity and the simplicity of its operations, as well as its industrial

applicability [7,8]. Most methods of adsorption use a wide range of materials, such as biological materials, mineral oxides, polymer resins, and activated carbon [7,9]. Activated carbon, especially, has found wide application in the adsorption process because of its large surface area, porosity, and capacity to adsorb contaminant; thus, it remains a very versatile and efficient adsorbent over many years of working experience [10].

Although commercial activated carbons with appropriate properties to remove heavy metals are widely available [11], Only a few apply to the efficient extraction of these elements, and they are usually expensive [12,13]. This economic constraint has motivated efforts in the study of low-cost and high-performance activated carbons using locally available and renewable materials [14]. Many works have shown the possibilities of agricultural waste and low-cost products like coconut shells [15], rice husk [16], pistachio and walnut shells [17], fruit seeds, sawdust [18], straw, and zeolites as feedstock in making activated carbon with high adsorption properties [19]. Walnut shells are a promising feedstock in this sense. They are an inexpensive and otherwise unused agricultural waste, and research shows that walnut-based activated carbon has the potential to perform better cadmium adsorption than commercially available products [20,21]. The objective of this study is to develop activated carbon derived from walnut shell agricultural waste and to investigate its effectiveness in removing cadmium ions (Cd^{2+}) from water and wastewater. This study aims to offer an economically feasible, long-term and ecologically viable solution to the problem of heavy metal purification by using a low-cost resource that is renewable, and can be recycled to a greater extent, engaging not only the problem of water quality but also the problem of the valorization of waste produced in agriculture.

2. Materials and methods

2.1. Chemical and instruments

In this research, an atomic absorption spectrophotometer (Shimadzu UV-1800, Kyoto, Japan) was used to determine the concentration of cadmium in the aqueous solutions.

A Nabertherm furnace model (LHT 02/17LB, Lilienthal, Germany) was used to prepare the adsorbent, and an Extech PH220 pH meter (Nashua, NH, USA) was used to measure the pH of the solutions. A shaker model (IKA MS 3 Basic, IKA, Breisgau, Germany) was used during the adsorption experiments to ensure that the solutions were uniformly mixed.

Cadmium chloride hydrate ($CdCl_2 \cdot H_2O$, Merck, Darmstadt, Germany) was used for the preparation of cadmium stock solutions. The solution pH was adjusted using dilute sulfuric acid and sodium hydroxide (Merck). Sodium thiosulfate and potassium iodide, also obtained from Merck, were applied in the preparation and characterization of the activated carbon material. Distilled water was used in all experimental procedures.

Walnut shells were selected as the raw precursor for activated carbon synthesis. The shells were ground and sieved to a particle size of 15–30 mesh and chemically activated with phosphoric acid. The raw material was impregnated with phosphoric acid at a mass ratio of 1:1 and mixed thoroughly, followed by the addition of distilled

water at two to three times the mixture weight to ensure proper dispersion. The slurry was dried in an oven at 110 °C for 24 h, then carbonized in the furnace at 490 °C with a heating rate of 8 °C/min and held at the target temperature for 45 min. After cooling, the carbon was repeatedly washed with distilled water, dried, and sieved to the desired particle size.

The prepared activated carbon was characterized to determine its physicochemical properties. The textural properties, including specific surface area and pore size distribution, were analyzed using the Brunauer–Emmett–Teller (BET) method, while porosity was measured with a NOVA Series 1000 porosimetry analyzer (Anton Paar QuantaTec, Boynton Beach, FL, USA). The iodine numbers of both the synthesized activated carbon (SAC) and commercial activated carbon (CAC) were determined. The main properties of the adsorbents are summarized in (**Table 1**). All experiments were conducted as single measurements without replicate runs; therefore, statistical analyses such as standard deviation and error estimation were not performed.

Table 1. Exhibit the physical properties of SAC and CAC.

Adsorbent	S_{BET} (m^2 /g)	Porosity	Particle size (mesh)	Iodine Number (mg/g)
SAC	960	64%	90-235	912
CAC	910	58%	80-230	618

2.2. Cd^{2+} adsorption tests

To make solutions with cadmium ions, cadmium chloride monohydrate ($CdCl_2 \cdot H_2O$) was dissolved in distilled water. First, a stock solution with a concentration of 1000 mg/L was made. Then, working solutions with the desired concentrations were made by diluting the stock solution. We put 0.2 g of activated carbon and 100 mL of cadmium solution at the right concentration into 150mL Erlenmeyer flasks for each adsorption experiment. The tests were done at a steady temperature of 30 °C and with constant stirring from a shaker set to 300 rpm. After completion of each experiment, the solutions were filtered, and the concentration of residual cadmium ions was measured using a Shimadzu AA-670 atomic absorption spectrophotometer. The amount of cadmium adsorbed by the carbon (q, mg/g) was calculated according to the **Equation (1)**.

$$q = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where C_0 is the initial cadmium concentration (mg/L), C_e is the equilibrium concentration (mg/L), V is the volume of the solution in (L), and m is the mass of the adsorbent in (g). Preliminary experiments were conducted to determine the equilibrium time for adsorption. To study the effect of the initial cadmium concentration, experiments were carried out within the range of 25–1000 mg/L, while other operating conditions, including temperature (30 °C), contact time, stirring speed (300 rpm), and pH (4.5), were kept constant.

Additional experiments were performed to investigate the effect of solution pH on adsorption efficiency. For this purpose, the pH was adjusted within the range of 1–12. All experiments were repeated using commercial activated carbon (CAC) under

the same conditions for comparison, with adsorbent characteristics as described earlier.

3. Results and discussion

3.1. Effect of time and pH

The influence of contact time on the adsorption of cadmium ions by the prepared activated carbon (SAC) is illustrated in (**Figure 1b**). Cadmium ions were quickly absorbed by the available active sites on the carbon surface during the early phases of the experiment, as demonstrated by the adsorption capacity increasing gradually over time. The presence of numerous initially unoccupied surface sites on the adsorbent accounts for the rapid cadmium ion uptake.

The rate of adsorption progressively dropped as the contact time increased, indicating that diffusion into the pores became the rate-limiting step as the active sites were gradually occupied. After about 4 h, the adsorption curve eventually reached a plateau at equilibrium. A dynamic balance between the cadmium ions adsorbed on the SAC surface and those still in the solution had been reached, as evidenced by the lack of a discernible increase in adsorption capacity beyond this point. These results led to the selection of an equilibrium contact time of 4 h for all ensuing experiments.

At this equilibrium point, the maximum adsorption capacity of SAC for cadmium from a solution with an initial concentration of 100 mg/L was determined to be 15.6 mg of Cd^{2+} per gram of adsorbent. These findings indicate that SAC is highly effective in adsorbing cadmium ions from water.

The effect of solution pH on the adsorption of cadmium ions onto activated carbon is presented in (**Figure 1a**). Among the parameters influencing adsorption efficiency, pH was found to be one of the most critical. As the pH of the solution increased, the adsorption capacity of the activated carbons also increased, reaching maximum values in alkaline conditions. The highest adsorption efficiencies were obtained at pH 12, where the prepared activated carbon (SAC) achieved an adsorption capacity of 81.9 mg/g, while the commercial activated carbon (CAC) showed a similar performance with 80.5 mg/g at an initial cadmium concentration of 100 mg/L.

This trend can be attributed to the effect of pH on cadmium ion speciation in solution and the surface charge of the adsorbent. Lower uptake occurs at low pH levels because the high concentration of hydrogen ions fiercely competes with cadmium ions for active adsorption sites. Adsorption is improved as pH rises because electrostatic repulsion decreases and more functional groups are available for cadmium binding. Cadmium may also start to precipitate as hydroxide species above a particular pH threshold, which would explain the observed rise in removal efficiency.

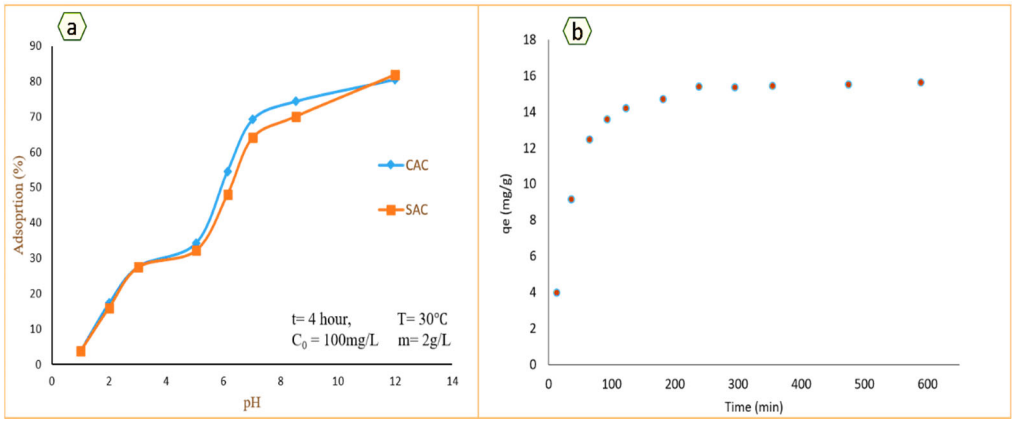

Figure 1. (**a**) shows the effect of pH on adsorption of cadmium by activated carbon, and (**b**) shows the effect of contact time on adsorption of cadmium ions.

3.2. Initial concentration effect

The effect of the initial cadmium ion concentration on the adsorption capacity of SAC is summarized in (**Table 2**). 56.4% of the cadmium ions were adsorbed at the lowest concentration tested (25 mg/g). The results show that as the initial concentration rises, the absolute amount of cadmium removed from the solution increases for a fixed amount of adsorbent. But, as concentration rises, the adsorption percentage falls. This behavior demonstrates how cadmium adsorption is highly dependent on the initial concentration of the solution. At low concentrations, the number of cadmium ions is relatively small compared to the available adsorption sites on the carbon surface, resulting in a high percentage of removal. As the initial concentration increases, the adsorbent surface sites become increasingly saturated, while the number of cadmium ions in solution continues to grow. Consequently, although more cadmium is adsorbed in absolute terms, the fraction of ions removed relative to the total present in the solution declines.

Table 2. Show the effect of initial concentration on Cd^{2+} adsorption on SAC and CAC.

C_0 (mg/L)	SAC(C_e)	SAC (q_e)	SAC (%)	CAC (C_e)	CAC (q_e)	CAC (%)
25	10.95	7.02	56.4	9.55	7.72	60.8
50	28.48	10.78	43.1	25/4	12.3	49.2
100	68.6	15.7	31.4	65.5	17.25	34.5
200	149.8	25.1	25.1	143.8	28.1	28.1
400	309.2	45.4	22.7	296.4	51.8	25.9
600	502.8	48.6	16.2	747.6	62.7	20.9
800	689.8	55.12	14.8	611.1	69.45	17.4
1000	873.6	63.2	12.6	847.6	76.2	15.2

3.3. Adsorption isotherms

Equilibrium adsorption isotherms represent a key concept in the study and optimization of adsorption systems. These isotherms are classified based on the slope of the initial segment of the adsorption curve, with H-type isotherms, as described in

the Giles classification, serving as a representative example [22]. The H-type isotherm is one of the most common isotherms available, in which there is a high affinity between the adsorbent and the adsorbate, and therefore there is no competition between the solvent and the solute [23]. The adsorption isotherms obtained on cadmium by two adsorbents, SAC and CAC, are shown in (**Figure 2a,b**), respectively.

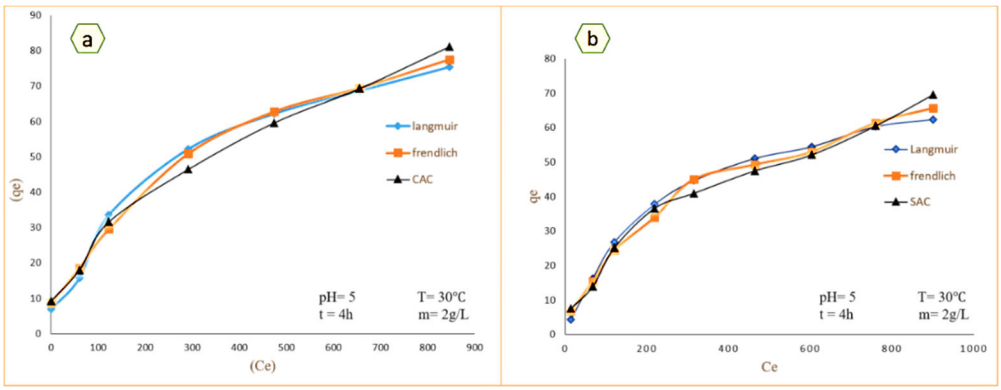

Figure 2. (**a**) Adsorption isotherms for cadmium ions on CAC. (**b**) Adsorption isotherms for cadmium ions on SAC, with a comparison and analysis against the Langmuir and the Freundlich models, Error bars are not shown as experiments were conducted without replicate measurements.

Langmuir Model: The fundamental premise of the Langmuir model is that adsorption takes place uniformly in a single layer across the active surface areas, with no interactions between the adsorbed molecules. In the case of single-layer adsorption, the Langmuir equation is expressed as **Equation (2)**.

$$q_e = \frac{Q^0 b C_e}{(1 + bC_e)} \tag{2}$$

And its linear form is as **Equation (3)**.

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} \tag{3}$$

Q_0 is the Adsorption capacity (mg/g), b is the Langmuir isotherm constant, and C_e and q_e also have the same definitions as before. From the results of experiments, the Langmuir isotherm curve is shown in (**Figure 3a**).

Freundlich Model: Freundlich model showed that the adsorption process is governed by the following equation:

whose logarithmic form is as **Equation (4)**.

$$q_e = K_f (C_e)^{1/n} \tag{4}$$

The logarithmic form is as **Equation (5)**.

$$ogq_e = logK_f + \frac{1}{n} logC_e \tag{5}$$

In the above equation, K_f and $1/n$ are the constants of the Freundlich equation.

Using the results of the experiments, the Freundlich adsorption isotherm for cadmium was plotted and shown in (**Figure 3b**).

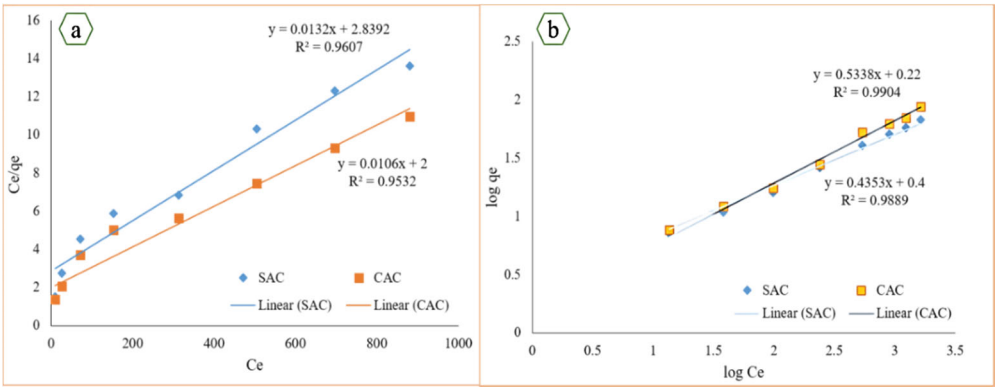

Figure 3. (**a**) Langmuir isotherm plots for SAC and CAC samples; (**b**) Freundlich isotherm plots for SAC and CAC samples.

The Freundlich and Langmuir constants were obtained from plotting the log q_e versus log C_e and C_e/q_e versus C_e plots, respectively, and are given in (**Table 3**) for both SAC and CAC carbon samples.

Table 3. Show the constants of Freundlich and Langmuir for SAC and CAC samples.

Adsorbent	Langmuir			Freundlich		
	Q_0 (mg/g)		R^2	K_f	1/*n*	R^2
SAC	75.7	0.0046	0.960	1.66	0.53	0.990
CAC	94.4	0.0053	0.953	2.51	0.43	0.988

According to the data obtained from (**Table 3**), it can be easily concluded that the adsorption of cadmium on CAC and SAC activated carbons had a better agreement with the Freundlich model.

4. Conclusion

This study successfully produced activated carbon from walnut shells and evaluSated its efficiency in removing cadmium ions from water, highlighting the potential of underutilized biomass as a low-cost adsorbent. The adsorption experiments demonstrated that the initial concentration of cadmium plays a critical role in removal efficiency, with higher concentrations leading to a decrease in adsorption due to the saturation of available active sites on the adsorbent surface. Furthermore, the impact of pH on cadmium adsorption was systematically investigated for both synthesized activated carbon (SAC) and commercial activated carbon (CAC), revealing that the adsorption capacity reached its maximum within a pH range of 7 to 12. This indicates that the surface properties of the activated carbon and the ionization state of cadmium are optimized in this pH range, facilitating stronger interactions and more efficient binding of cadmium ions. The equilibrium adsorption data were analyzed using both the Langmuir and the Freundlich isotherm models, with the Freundlich model providing a slightly better fit, suggesting the presence of surface heterogeneity and multiple adsorption energies. These findings imply that adsorption on walnut shell-derived activated carbon is influenced by a combination of surface

properties and solution chemistry, which can guide the optimization of operational parameters in practical water treatment applications.

Author contributions: RJ and MA conceptualized and supervised the study; NM, RJ and AR collected the row data; R.J. wrote the manuscript with input from all authors. All authors reviewed and approved the final version.

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