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RESEARCH PAPER

Adsorptive Removal of Copper Ions from Water by HCl-Activated Coconut Shell Carbon with Micro/Nanoscale Surface Features

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ABSTRACT

Activated carbon as a micro/Nano surface, a cost-effective adsorbent derived from coconut shells, was utilized to remove Cu(II) ions from aqueous solutions. The preparation process of activated carbon involves using 0.1 N hydrochloric acid followed by carbonization. Several techniques, including FESEM, TEM, and XRD, characterized the activated carbon. The activated carbon exhibited a network of cavities and a mesoporous structure, characterized by a rough surface with irregularly sized pores. Some pores were blocked by carbon and other impurities, such as silicon. The activated carbon's surface chemistry exhibited various functional groups associated with acidic properties. The adsorption capacity for Cu(II) ions was significantly influenced by factors such as the initial pH of the solution, concentration, contact time, and the mass of the adsorbent. Indicate that as the activated carbon weight increased from 0.01 to 0.1 g, the adsorption capacity for Cu(II) gradually decreased, while the removal rate increased significantly. Endothermic adsorption involves an increase in adsorption that is directly proportional to temperature, resulting from the rise in the number of adsorption sites with increasing temperature. Investigations into equilibrium revealed a Freundlich isotherm model, with R^2 values greater than 0.9654. This finding indicates that coconut shells can be effectively transformed into activated carbon, which is suitable for removing Cu(II) ions from aqueous solutions.

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INTRODUCTION

Pollution is the introduction of harmful substances into the environment, and these toxic substances are known as pollutants. Pollutants, such as volcanic ash, can occur naturally or be generated by human activities, including garbage and runoff from factories. These pollutants negatively impact the quality of air,

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water, and land. Water pollution occurs explicitly when harmful substances—often chemicals or microorganisms—contaminate bodies of water, such as streams, rivers, lakes, oceans, or aquifers. This contamination degrades water quality and can make it toxic to humans and the environment. Water pollution is a significant global issue that affects populations worldwide [1]. Daily, large quantities of wastewater are generated from industrial, agricultural, and domestic activities, which are often discharged into the ground or water bodies. The widespread introduction of pollutants leads to water pollution on a global scale. Therefore, purifying contaminated wastewater before returning it to water bodies or land is crucial. Various pollutants contribute to environmental pollution, including heavy metals, radionuclides, phenols, pesticides, and other substances [2-4]. Heavy metals, in particular, are a significant class of pollutants produced in large quantities by industries. They pose a serious threat to many aquatic organisms and human health due to their toxicity and potential carcinogenic effects. Among the proven methods for removing dyes from wastewater, such as absorption, coagulation, advanced oxidation, and membrane separation, absorption is the most effective advanced wastewater treatment process, yielding satisfactory results [5, 6].

Adsorption is considered the most important and widely used method. Due to its low cost and ease of use, it holds great promise for removing the most dangerous contaminants, including metal ions. Several adsorbent materials, including bio-clay, silica, and activated carbon, have been used previously. Adsorption typically relies on the interaction of the adsorbent's functional groups with the metal ions, which significantly impacts efficiency, capacity, selectivity, and regeneration potential [5-11].

Heavy metal ion pollution from industries, including plastics, printing, paper, and textiles, is a significant problem that threatens human health and all living organisms, and also disrupts the ecological balance. Therefore, various methods, including coagulation, catalytic decomposition, oxidation, and adsorption, are employed to remove heavy metal ions from wastewater. Among the most important of these methods, adsorption stands out due to its ease of use, implementation, and design [12, 13]. Water pollution caused by heavy metal ions poses a significant threat to the environment and the health of aquatic organisms due to their toxicity and non-biodegradability. Cu^{2+} is commonly found in drinking water and industrial wastewater from various textile industries, including mining, metal plating, and the production of printed circuit boards, as well as pipe and metal corrosion. However, the improper disposal of industrial wastewater containing Cu^{2+} can lead to health problems for aquatic organisms and cause severe environmental issues. [14-17].

In this work, we converted coconut shells into activated carbon to remove Cu(II) from aqueous solutions. The resulting AC was analyzed using XRD, TEM, and FESEM. The effects of ion concentration, AC weight, and temperature on the adsorption efficiency are investigated, and the adsorption properties of the AC are also evaluated using Cu^{2+} as the model ion.

MATERIALS AND METHODS

Preparation of Hydrochloric Acid-Activated Carbon from Coconut Shells (AC-HCl)

Coconuts were obtained from a local supermarket in Iraq. The shells were thoroughly washed with distilled water to remove any adhering impurities. Only the shells were retained for use after manually separating and discarding the pulp. These shells were sun-dried, ground, and sieved for three days to obtain a fine coconut shell powder. The activation process involved two primary steps. First, the dried coconut shell powder was carbonized at 500 °C for two hours in an oxygen-free environment to produce biochar. After cooling to room temperature, the resulting carbon was immersed in a 50% hydrochloric acid solution prepared with 100 mL of distilled water and allowed to soak for 24 hours. Following the acid treatment, the sample was repeatedly washed with distilled water until the pH of the filtrate became neutral. Finally, the activated carbon was dried to a constant weight and stored in a clean, dry, airtight glass container for subsequent use. As shown in Fig. 1:

Determination of heavy metals

To determine the capacity of the activated carbon to remove heavy metal ions, 0.05 g of the AC-HCl was added to solutions containing copper (II) ions. All adsorption experiments were conducted in a temperature-controlled shaker operating at 160 rpm and a temperature of 25 °C. The concentration of remaining metal ions in the

solution was measured using atomic absorption spectroscopy. The removal percentage and the amount of adsorbed metal ions (Q, in mg/g) were calculated using the following Eqs. 1 and 2:

$$E\% = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

$$Q_e = \frac{(C_o - C_e)V}{m(g)} \quad (2)$$

RESULTS AND DISCUSSION

Characterization for adsorbent/adsorbate

X-ray Diffraction (XRD) Analysis of Activated Carbon (AC)

X-ray diffraction (XRD) is a powerful technique used to investigate nanomaterials' crystalline structure, phase purity, and various microstructural properties. It provides valuable information regarding lattice parameters, crystallite size, lattice strain, structural defects, and the nature of chemical bonding within the crystalline phases. Fig. 2 presents the prepared activated carbon (AC) XRD pattern. The pattern exhibits two broad diffraction peaks centered at 2θ = 25.82° and 42.22°, which are characteristic of the (002) and (100) planes of amorphous or turbostratic carbon structures, respectively[18]. The broad nature of these peaks suggests a low degree of crystallinity and the presence of disordered graphitic domains within the carbon matrix. These features are typical for activated carbon materials, indicating successful carbonization and partial graphitization, with high surface area and structural irregularities favorable for adsorption applications. [19-21].

Field Emission Scanning Electron Microscopy (FESEM) Analysis of Activated Carbon (AC)

Field Emission Scanning Electron Microscopy (FESEM) was utilized to investigate the surface morphology of activated carbon (AC) before and after the adsorption of Cu(II) ions. As shown in Fig. 3(a), the FESEM micrograph of the AC prior to adsorption reveals an irregular, rough surface morphology with the absence of any visible cross-linking among the AC particles. The presence of small white aggregates dispersed across the surface is attributed to the chemical activation process with hydrochloric acid (HCl), which likely

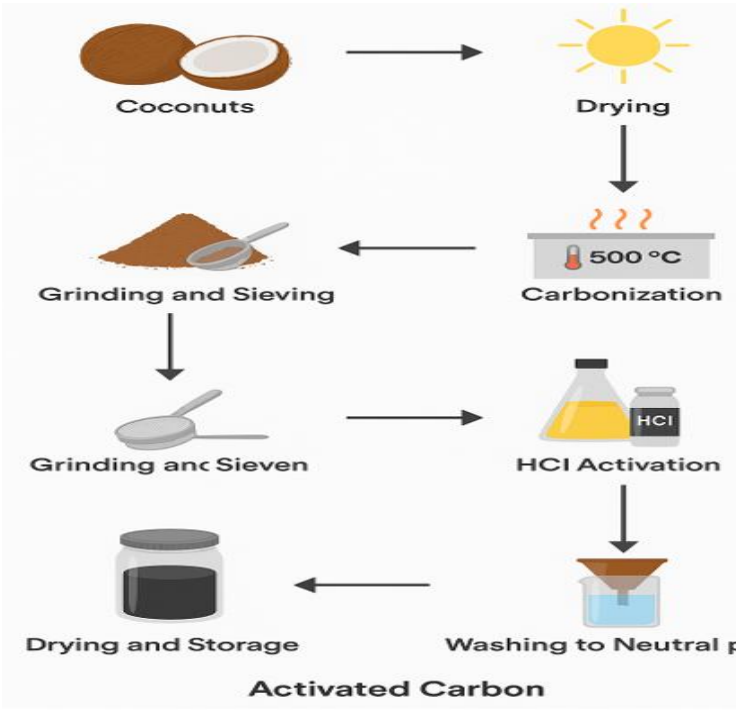

Fig. 1. Preparation of Hydrochloric Acid-Activated Carbon from Coconut Shells (AC-HCl).

introduced microstructural irregularities and enhanced the porosity. The surface morphology underwent a noticeable transformation following the adsorption process, as illustrated in Fig. 3(b). The AC surface exhibited a more defined sheet-like structure with increased porosity, which can be attributed to both acid activation and the interaction with Cu(II) ions. These ions may have facilitated surface cross-linking or deposition, contributing to the observed morphological changes. Additionally, the surface displayed a denser arrangement of rough granules and particles, further confirming the involvement of Cu(II) during the adsorption process [22-24].

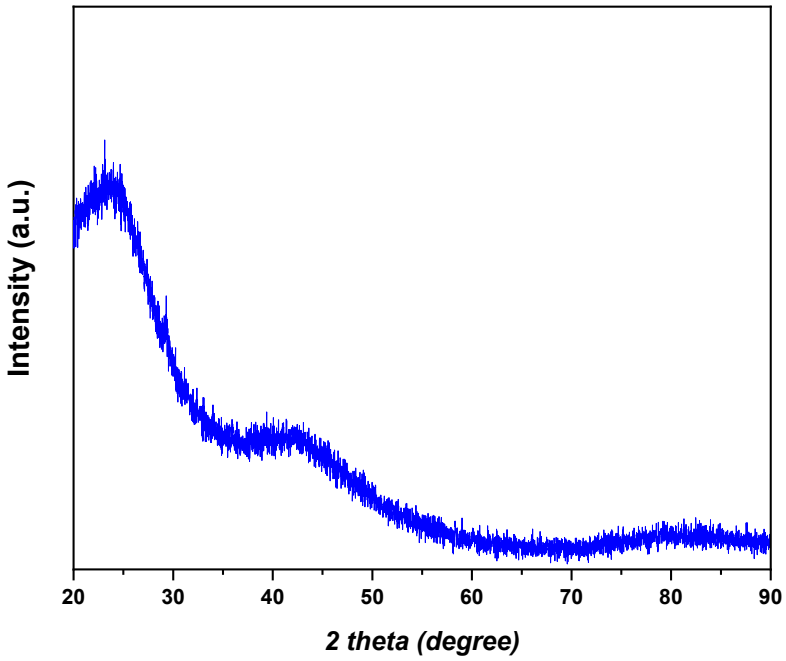

Fig. 2. X-ray diffraction (XRD) analysis of activated carbon Micro/Nano surface.

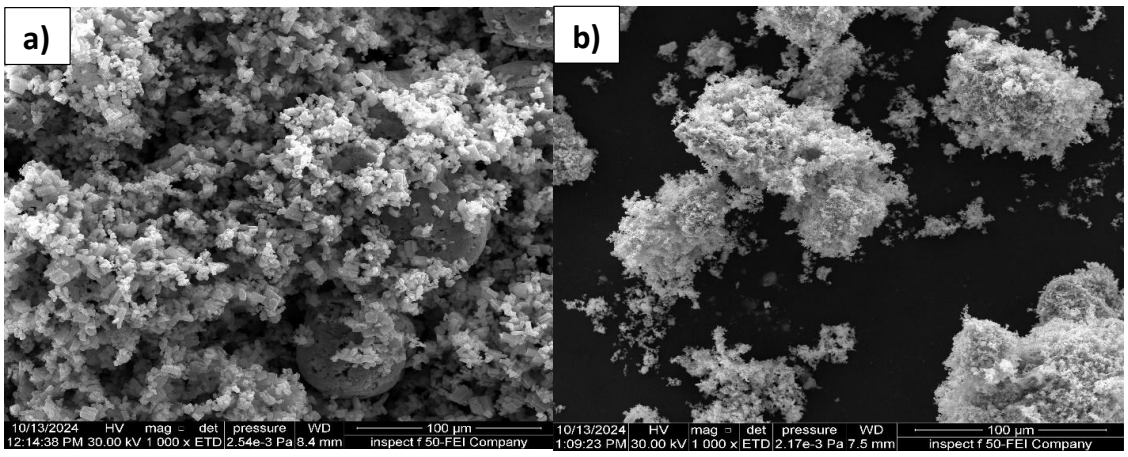

Fig. 3. ESEM image of a) activated carbon Micro/Nano surface before adsorption, b) after adsorption.

Transmission Electron Microscopy (TEM)

TEM surface analysis was conducted to examine the morphology of the activated carbon surface. As illustrated in Fig. 4, the surface appeared more accessible, and a new geometric structure formed after acid was decorated onto the activated carbon. This change may be attributed to the presence of acid on the surface. Fig. 4 also reveals that the architecture resembles a structure composed of many single-crystal plates, along with disordered, wormhole-like pores within the particles, indicating the presence of a mesoporous

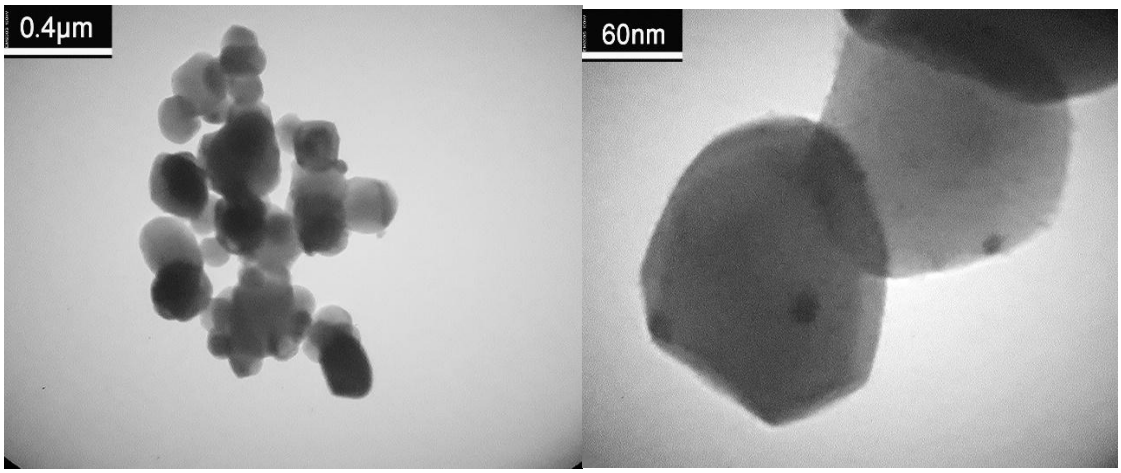

Fig. 4. TEM image of activated carbon Micro/Nano surface.

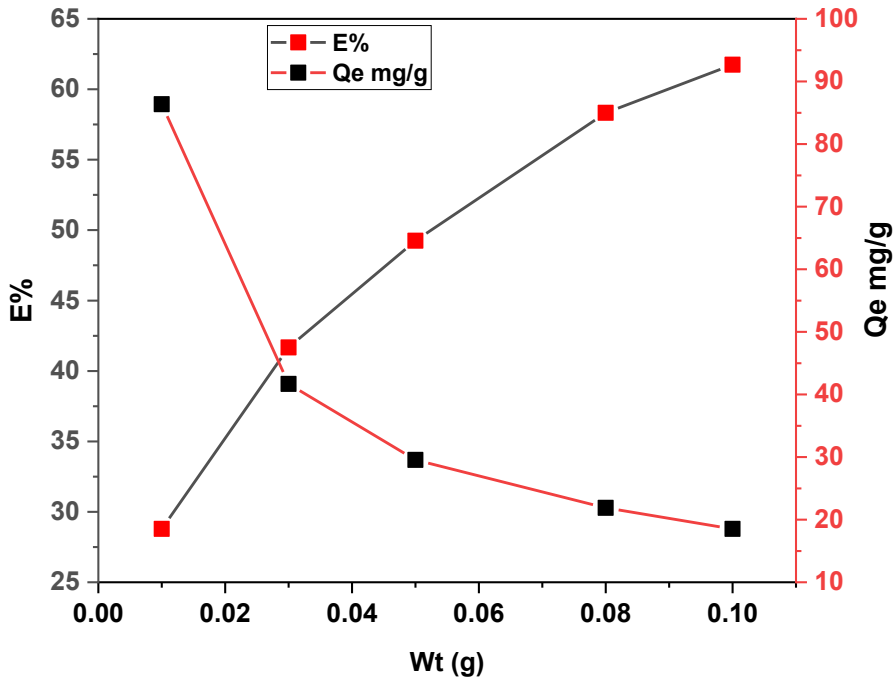

Fig. 5. Effect of the weight of the activated carbon Micro/Nano surface on the removal of Cu(II)ion.

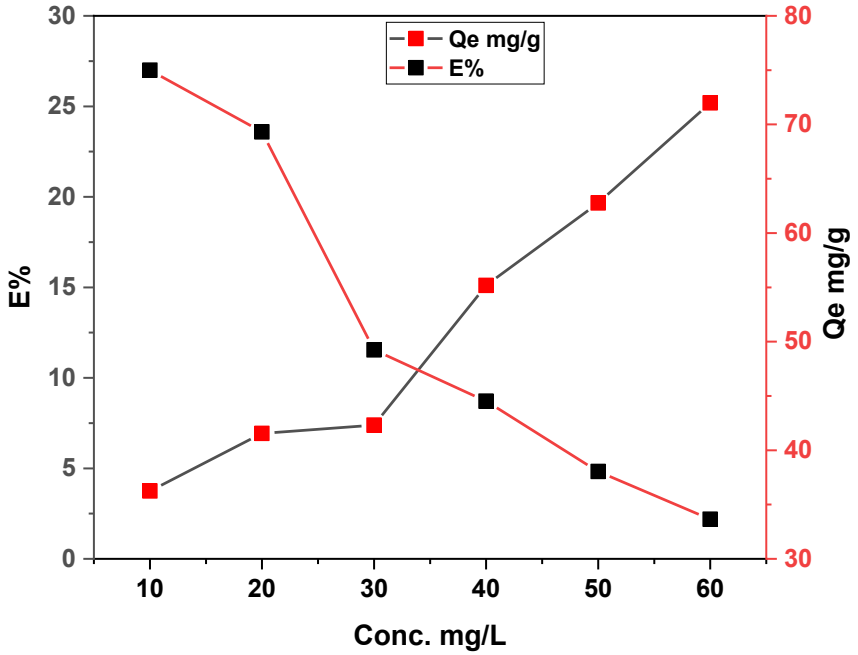

Fig. 6. Effect of the concentration of Cu (II) solution by using activated carbon Micro/Nano surface.

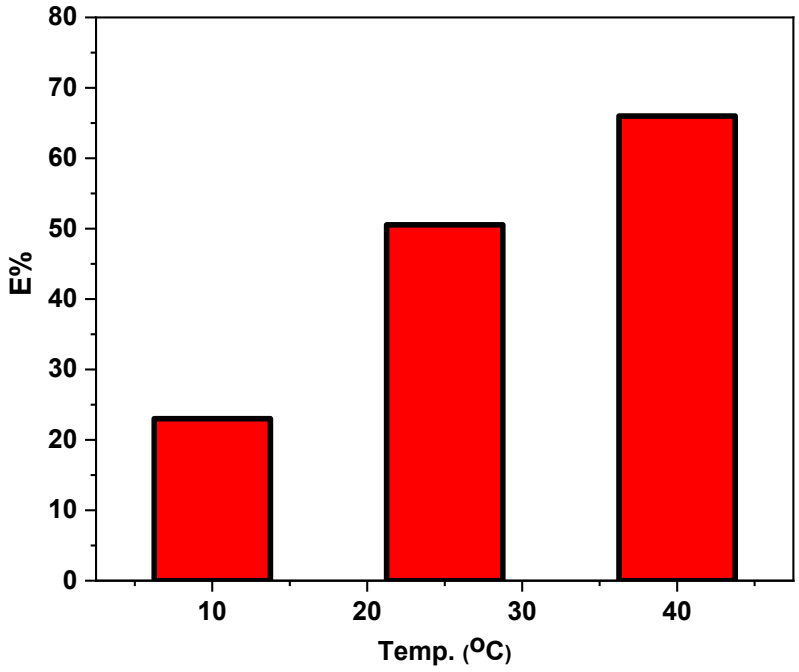

Fig. 7. Effect of Temperature solution on the removal of Cu(II) ions by activated carbon Micro/Nano surface.

structure. The average size observed in the spherical TEM images of the activated carbon was between 0.40µm and 60 nm [13, 25].

Effect of the weight of the activated carbon

A series of activated carbon weights ranging from 0.01 to 0.1 g was added to a solution containing 30 mg/L of Cu(II). The mixture was shaken, and the adsorption rate reached equilibrium at 25 °C. The pH of 100 mL of the 30 mg/L Cu(II) solution was adjusted to 7.0. We examined the effect of varying amounts of activated carbon on the adsorption of Cu(II). The results, shown in Fig. 5, indicate that as the activated carbon weight increased from 0.01 to 0.1 g, the adsorption capacity for Cu(II) gradually decreased, while the removal rate increased significantly. When the activated carbon weight reached 0.1 g, the Cu(II) removal rate stabilized and ceased fluctuating. As the weight of activated carbon increases, the number of active sites available for Cu(II) adsorption also increases. In contrast, the adsorption efficiency decreases as the weight of activated carbon increases [26, 27] .

Effect of the Cu(II) concentration

In this study, 100 mL of Cu(II) solutions with concentrations ranging from 10 to 60 mg/L were adjusted to a pH of 7.0. Following this adjustment, 0.05 g of activated carbon was added to the Cu(II) solutions and mixed at 25 °C. The focus of the study was to investigate how different initial Cu(II) concentrations affected the adsorption of Cu(II) by the activated carbon. The results, illustrated in Fig. 6, demonstrate that as the Cu(II) concentration increased from 10 mg/L to 60 mg/L, the adsorption efficiency of the activated carbon for Cu(II) varied. Specifically, the efficiency decreased from 2.45 ± 0.05 mg/g to 0.244 ± 0.69 mg/g. Initially, the removal rate remained stable but gradually reduced as the concentration increased. At lower concentrations of Cu(II) ions, the active carbon does not reach adsorption saturation and can effectively remove the ions from the solution. However, at higher concentrations, all active adsorption sites on the active carbon surface become saturated, decreasing the removal rate as the initial Cu(II) concentration increases [28, 29].

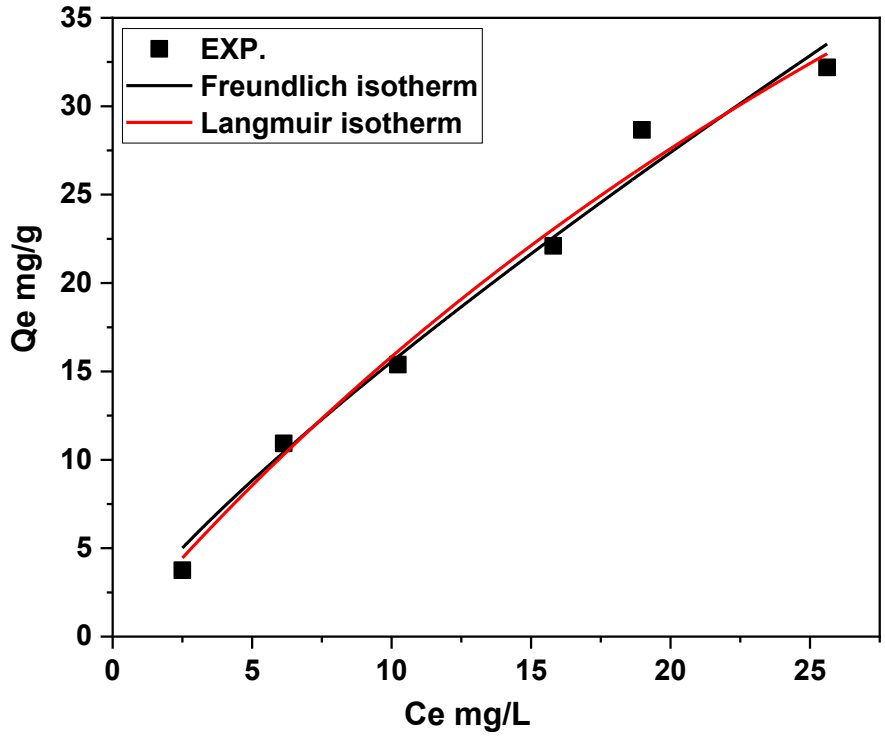

Fig. 8. Adsorption isotherm Freundlich model, and Langmuir model for Copper ions on the activated carbon Micro/Nano surface.

Effect of Temperature

To determine whether the adsorption process is endothermic or exothermic, adsorption curves were calculated for various copper (II) ion systems on the adsorbent surface. We then examined the removal of copper ions at different temperatures (10, 25, and 40°C) in the presence of varying ions concentrations (30 mg/L), as shown in Fig. 7. The results indicated an equilibrium adsorption capacity for copper ions, showing that the adsorption efficiency of the adsorbent changes with increasing temperature. Therefore, temperature is a key factor in both chemical and physical processes. Endothermic adsorption involves an increase in adsorption that is directly proportional to temperature, resulting from the rise in the number of adsorption sites with increasing temperature. It has been found that increasing temperature causes a decrease in the viscous forces of the aqueous phases, leading to faster diffusion of ions into the solution. The removal process is also significantly affected by changes in the solubility of the adsorbent molecules. In some cases, the enlargement of pore size at high temperatures also leads to an increase in adsorption[7, 30, 31].

Adsorption Isotherm

The Cu(II) removal experiment results were analyzed using several isothermal models, specifically the Langmuir and Freundlich models. The mathematical equations for these models, which describe the isothermal adsorption of Cu(II) onto activated carbon, are presented in Eqs. 3 and 4. The findings indicate a better fit with the Freundlich model, which is appropriate for heterogeneous surfaces and effectively describes multilayer adsorption. In contrast, the Langmuir model assumes that adsorption occurs on a homogeneous, single-layer surface without interactions between adsorbent molecules [20, 32].

The Langmuir adsorption isotherm effectively explains the adsorption of Cu(II) from aqueous solutions. Equation (3) provides the expression for the Langmuir model.

$$Q_e = \frac{Q_m K_L C_e}{1+K_L C_e} \quad (3)$$

The Freundlich adsorption isotherm is an empirical equation employed to describe heterogeneous systems. The Freundlich equation is expressed as:

$$Q_e = K_f C_e^{\frac{1}{n}} \quad (4)$$

Where qe (mg/g) is the amount of adsorbed Cu(II) per unit mass of sorbent and Ce (mg/L) adsorbed Cu(II) concentration in solution at equilibrium, a Qmax is the maximum amount of the Cu(II) .K_L: constant denoting the energy of

Table 1. Two Parameters of adsorption isotherm models of Langmuir and Freundlich.

Isotherm		Value
Freundlich	K_f	2.377
	1/n	0.244
	R^2	0.9789
Langmuir	q_m (mg/g)	35.55
	K_L (L/mg)	0.155
	R^2	0.9421

adsorption and affinity of the binding sites (L/mg), K_f: Freundlich constant (mg/g) (L/mg), n: adsorption intensity[33].

The Freundlich model provides a good fit to the experimental data, as indicated by a correlation coefficient (R^2) of 0.9789, as illustrated in Fig. 8 and detailed in Table 1. In contrast, the Langmuir isotherm shows poor agreement with the experimental data, with a correlation coefficient (R^2) of less than 0.9421. According to the Freundlich model, the multilayer adsorption capacity at 25 °C was measured at 33.33 mg/g. The reliability of the Freundlich equation, which assumes a heterogeneous surface, suggests that the excellent fit of the Freundlich isotherm to the experimental data may be due to a varied distribution of active sites on the activated carbon. This finding aligns with previous research on drug sorption across different sorbents. For example, in the study of methylene adsorption on oil palm fiber-activated carbon, the Freundlich isotherm was similarly applicable [34, 35]

CONCLUSION

This study aimed to explore the equilibrium aspects of adsorption processes. To enhance efficiency and increase the number of active sites, we used practical, inexpensive, readily available, and nontoxic adsorbents, such as activated carbon derived from natural materials like coconut shells. Activated carbon, recognized for its environmentally friendly properties, can be prepared from these readily available sources. To further improve the environmental suitability of activated carbon, acid activation is employed to increase its surface area. As the weight of activated carbon increases, the number of active sites available for Cu(II) adsorption also increases. In contrast, the adsorption efficiency decreases as the weight of activated carbon increases. At lower concentrations of Cu(II) ions, the activated carbon does not reach adsorption saturation and can effectively remove the ions from the solution. However, at higher concentrations, all active adsorption sites on the activated carbon surface become saturated, resulting in a decrease in the removal rate as the initial Cu(II) concentration increases. We conducted experiments to remove copper(II) ions from aqueous solutions through adsorption, achieving promising results at a temperature of 25°C using 0.05 g of activated carbon.

CONFLICT OF INTEREST

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

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