

Comparative Study on Cu(II) Adsorption Performance of Self-made and Commercial Activated Carbon

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Abstract: *Using coconut shell particles as raw material, coconut shell activated carbon was prepared by first carbonizing the particles and then applying chemical activation. The adsorption of copper ions from wastewater by coconut shell activated carbon was investigated, with coconut shell activated carbon and copper ions as the research objects. Experimental instruments including scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and ultraviolet-visible spectrophotometry (UV-vis) were employed to analyze the activated carbon and its adsorption performance. The results show that coconut shell activated carbon prepared by carbonization followed by phosphoric acid activation exhibits significantly better Cu(II) adsorption performance than samples activated directly without carbonization and than commercial activated carbon. Under the conditions of a solid-to-liquid ratio of 1:2, an activation temperature of 600 °C, and an activation time of 1 h, the carbonized activated carbon performs best, achieving an adsorption rate of 91.2% and an adsorption capacity of 18.24 mg/g for a 100 mg/L Cu(II) solution. FTIR analysis confirms that after adsorption, the peak positions of oxygen-containing functional groups such as hydroxyl and carboxyl groups shift, and a characteristic Cu—O vibration peak appears, indicating that chemisorption and surface complexation are the dominant adsorption mechanisms. The optimal adsorbent dosage is 0.5 g/L. This study provides a theoretical basis and technical reference for the resource utilization of agricultural and forestry wastes and the low-cost purification of copper-containing wastewater.*

Keywords: Coconut shell activated carbon; Phosphoric acid activation; Copper ions; Adsorption performance; Solid-to-liquid ratio; Dosage.

1. INTRODUCTION

With the continuous expansion of industrial production and daily human activities, the direct discharge of inadequately treated domestic and industrial wastewater has led to increasingly severe heavy metal pollution in water bodies. Mining, metal smelting, mechanical processing, and electroplating industries generate large volumes of heavy metal wastewater, predominantly containing copper, mercury, and silver ions [7,12]. Copper ions are highly toxic and non-biodegradable; excessive copper in water disrupts aquatic ecological balance and, through bioaccumulation in the food chain, damages the liver and hematopoietic system in humans, with chronic exposure potentially inducing chronic poisoning [6]. Therefore, the harmless treatment of copper-containing wastewater is a key research direction in water pollution control.

Current heavy metal wastewater treatment technologies include chemical precipitation, membrane separation, ion exchange, and adsorption. Among these, the adsorption method offers industrial application potential due to its simple operation, low operating cost, absence of secondary pollution, and recyclability [7,12]. Activated carbon, with its large specific surface area, well-developed pore structure, and tunable surface chemistry, is a mainstream high-efficiency adsorption medium [8]. The adsorption performance of activated carbon varies significantly depending on the biomass precursor. Researchers have utilized peanut shells [5], straw [3,12], waste fiberboard [6], *Ganoderma lucidum* bran [1], and palm fibers [10] to prepare activated carbon for heavy metal ion adsorption. As an abundant and low-cost agricultural and forestry waste, coconut shells possess a natural lignocellulosic structure that facilitates the construction of hierarchical micro-mesoporous channels. The resulting coconut shell activated carbon features low ash content, high mechanical strength, and a large number of tunable oxygen-containing active sites on the surface, giving it outstanding advantages in heavy metal ion adsorption [9,13,14]. Coconut-based biomass materials such as coir activated carbon fibers have also been used in copper ion adsorption studies [2]. Current research on coconut shell activated carbon mostly focuses on a single activation process [9,11,14], lacking systematic analysis of the carbon structure and copper ion adsorption performance. In this study, coconut shell carbonized material was used as a precursor to prepare modified coconut shell activated carbon via chemical activation. Microscopic characterization was employed to analyze the regulatory mechanisms of different activation processes on the morphology, pore volume, pore size, and surface functional groups of the activated

carbon. Static adsorption experiments were conducted to investigate the adsorption of copper ions from water under various environmental conditions, and the mechanism of copper ion removal by coconut shell activated carbon was elucidated. This work provides a theoretical basis and technical reference for the resource utilization of agricultural and forestry wastes and the low-cost purification of domestic and industrial copper-containing wastewater.

2. EXPERIMENTAL

2.1 Carbonization and Activation

Coconut shell raw material was placed in a vacuum tube furnace and subjected to high-temperature carbonization under nitrogen as a protective gas at 600 °C for 2 h [9,14]. After natural cooling, coconut shell carbonized material was obtained. The carbonized material was thoroughly mixed with phosphoric acid at solid-to-liquid ratios of 1:1, 1:2, and 1:3 (g/mL), then placed in a muffle furnace for high-temperature activation at 600 °C for 1 h [8]. After natural cooling, the activated carbon was washed with water to remove residual phosphoric acid, dried, and stored for later use.

2.2 Adsorption Experiments

A certain amount of activated carbon was mixed with 50 mL of copper ion solution at various concentrations, and adsorption was carried out in a thermostatic water-bath shaker at an oscillation frequency of 100 r/min. The supernatant was collected, filtered, and the copper ion concentration was measured. The adsorption rate R and adsorption capacity q_e were calculated using Equations (1) and (2).

$$R = (\rho_0 - \rho_e) / \rho_0 \times 100\% \quad (1)$$

$$q_e = (\rho_0 - \rho_e)V / m \times 100 \quad (2)$$

where ρ_0 is the initial mass concentration of the copper ion solution (mg/L); ρ_e is the equilibrium mass concentration of the copper ion solution (mg/L); R is the equilibrium adsorption rate of copper ions (%); q_e is the equilibrium adsorption capacity (mg/g); V is the volume of the copper ion solution (mL); and m is the adsorbent dosage (g).

2.3 Characterization

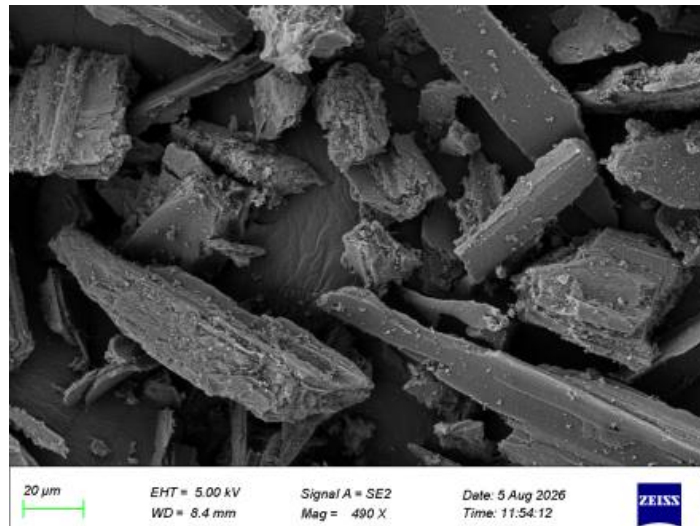
Scanning electron microscopy (SEM) was used to characterize the microscopic morphology of the prepared activated carbon. Fourier transform infrared spectroscopy (FTIR) was employed to analyze the functional groups of the activated carbon before and after adsorption [7,19]. An ultraviolet-visible spectrophotometer (UV-vis) was used for atomic absorption spectrometric determination of copper ion content in the solutions.

3. RESULTS AND DISCUSSION

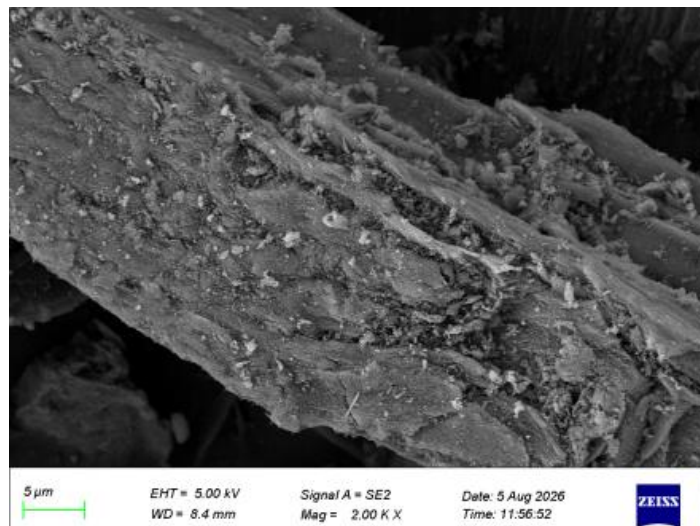
3.1 Characterization Analysis

3.1.1 SEM Morphological Analysis

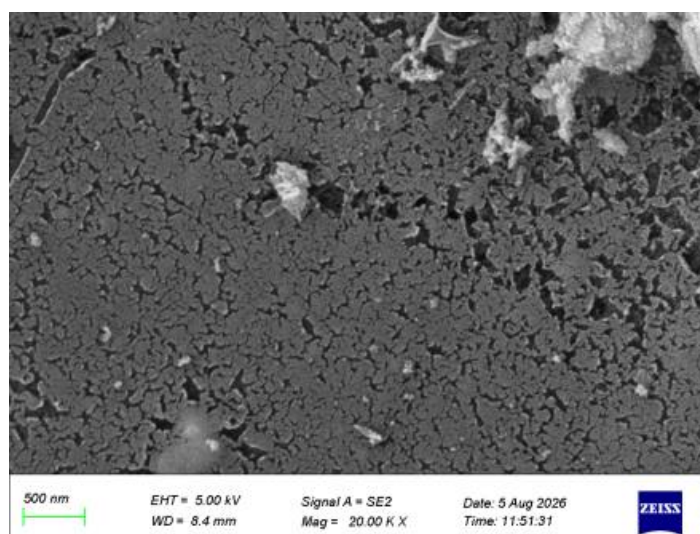
Scanning electron microscopy (SEM) was used to characterize the surface microstructure of the prepared coconut shell activated carbon, and SEM images at different magnifications are shown in Figure 1. From the low-magnification SEM image (500×), it can be seen that the coconut shell activated carbon prepared by phosphoric acid activation exhibits irregular flake, strip, and granular structures with uneven particle size distribution, mostly in the range of 10–50 μm. The activated carbon surface is rough and retains some fibrous structural characteristics of the coconut shell raw material (see Figure 1-a), which is attributed to the carbon skeleton structure formed by the decomposition of lignin and cellulose during high-temperature carbonization [9,14].



(a) 500×



(b) 2000×



(c) 20000×

Figure 1: SEM images of coconut shell activated carbon at different magnifications

At 2000 $\times$ magnification, a large number of pores and uneven structures can be observed distributed on the activated carbon surface (see Figure 1-b). During phosphoric acid activation, phosphoric acid penetrates into the interior of the coconut shell carbonized material as an activating agent and forms abundant pore structures on the carbon skeleton through etching. Phosphoric acid reacts with carbon at high temperatures, promoting pore formation and development through dehydrogenation and polycondensation. As an activating agent, phosphoric acid effectively etches the carbon matrix and constructs abundant microporous and mesoporous structures [8,11].

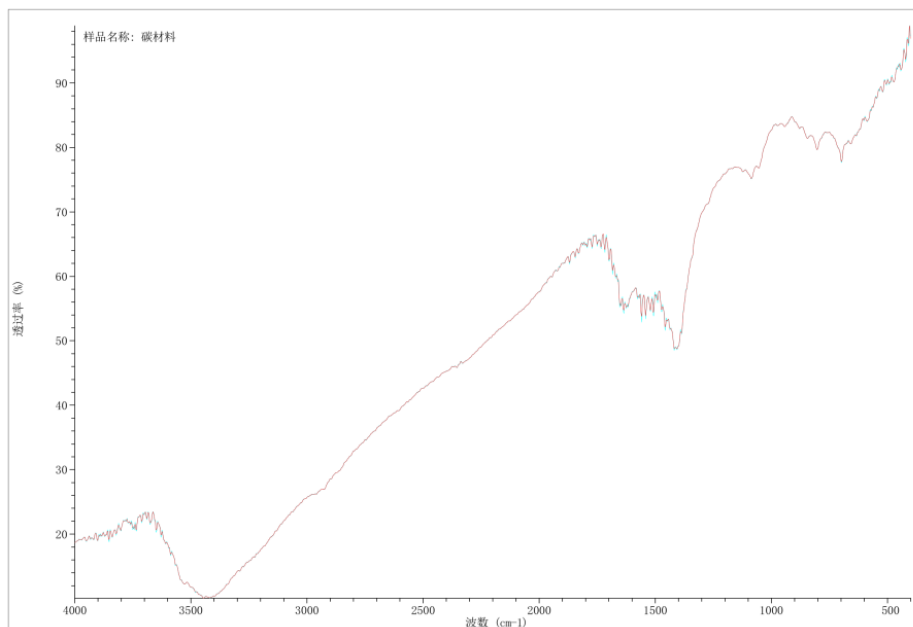
High-magnification SEM images (10000 $\times$ –20000 $\times$) further reveal the fine pore structure of the activated carbon (see Figure 1-c). A large number of nanoscale micropores and mesopores with varying sizes are distributed on the surface, exhibiting a hierarchical pore structure. These dense pore structures provide abundant adsorption sites for copper ion adsorption, while the well-developed mesoporous channels facilitate the diffusion and mass transfer of copper ions into the internal micropores of the activated carbon [4,5]. Additionally, obvious wrinkles and cracks are visible on the activated carbon surface, which further increase the specific surface area and facilitate the capture and adsorption of copper ions from water.

3.1.2 FTIR Spectroscopic Analysis

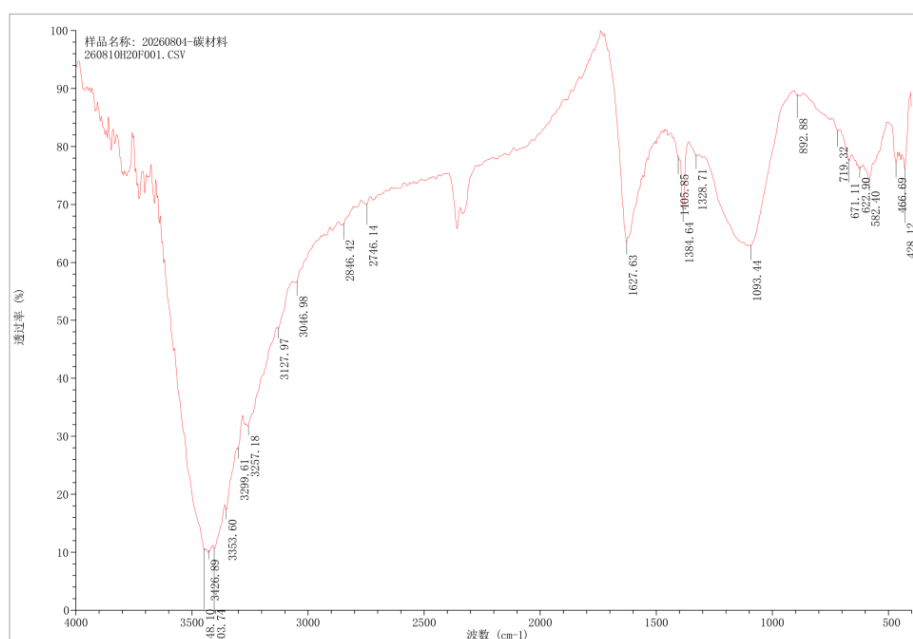
Fourier transform infrared spectroscopy (FTIR) was used to characterize the surface functional groups of phosphoric acid-activated coconut shell activated carbon before and after copper ion adsorption, and the spectra are shown in Figure 2. From the infrared spectrum before adsorption, a broad and strong absorption peak appears at 3400–3500 cm^{-1} , attributed to the stretching vibration of surface hydroxyl groups (—OH), indicating the presence of abundant hydroxyl groups and adsorbed water molecules on the activated carbon surface [7,15]. A weak absorption peak near 2850–2950 cm^{-1} corresponds to the asymmetric stretching vibration of aliphatic C—H bonds. The absorption peak at 1620–1640 cm^{-1} is attributed to the aromatic C=C skeleton vibration and the C=O stretching vibration in carboxyl groups [16]. The peaks in the 1380–1450 cm^{-1} region are attributed to the in-plane bending vibration of O—H and the symmetric stretching vibration of carboxylate (—COO^-). The strong absorption peak at 1000–1100 cm^{-1} corresponds to the stretching vibrations of C—O—C , C=O , and phosphate ester P—O—C bonds, which is closely related to the introduction of phosphorus groups during phosphoric acid activation [8,17]. The weak peaks in the 500–800 cm^{-1} region are attributed to the out-of-plane bending vibration of aromatic C—H .

After copper ion adsorption, the infrared spectrum undergoes significant changes. First, the hydroxyl stretching vibration peak in the 3200–3500 cm^{-1} region splits from a broad peak into multiple relatively sharp sub-peaks (3448, 3426, 3403, 3353 cm^{-1} , etc.), and both the peak shape and intensity change, indicating that hydroxyl oxygen atoms undergo coordination complexation with Cu^{2+} , altering the hydrogen bonding environment [15,18]. Second, the C=O/C=C peak at 1627 cm^{-1} shifts and decreases in intensity, indicating that carboxyl and carbonyl groups participate in the copper ion adsorption process, possibly binding to Cu^{2+} through surface complexation or ion exchange mechanisms [16]. Third, the intensity and position of the C=O/P—O—C absorption peak at 1093 cm^{-1} change, suggesting that oxygen atoms in phosphate groups also participate in the chelation of Cu^{2+} [17].

Most importantly, after adsorption, a series of new characteristic absorption peaks appear in the low-frequency region of 400–700 cm^{-1} , including 719, 671, 622, 582, 466, and 428 cm^{-1} . These peaks can be attributed to the stretching vibration of Cu—O bonds and the characteristic vibrations of copper-oxygen complexes [15,19], directly proving that Cu^{2+} has been successfully loaded onto the activated carbon surface and formed chemical bonds with oxygen-containing functional groups. The above infrared spectral changes indicate that the adsorption of copper ions by phosphoric acid-activated coconut shell activated carbon is not simply physical adsorption, but rather a chemisorption process with oxygen-containing functional groups such as hydroxyl, carboxyl, and phosphate ester groups as active sites, with surface complexation and ion exchange as the dominant mechanisms [7,12].



(a) Before adsorption



(b) After adsorption

Figure 2: FTIR spectra of coconut shell activated carbon before (a) and after (b) Cu(II) adsorption

3.2 Adsorption Experimental Results and Discussion

3.2.1 Comparison of Adsorption Performance Between Uncarbonized and Carbonized Raw Materials Activated with Phosphoric Acid

To investigate the effect of carbonized material pretreatment on the adsorption performance of activated carbon, uncarbonized coconut shell raw material and coconut shell carbonized material (carbonized at 600 °C for 2 h) were used as precursors. Phosphoric acid activation was performed at a solid-to-liquid ratio of 1:2, an activation temperature of 600 °C, and an activation time of 1 h to prepare two activated carbon samples. Their copper ion adsorption effects were compared under identical adsorption conditions (initial Cu^{2+} concentration of 100 mg/L, solution volume of 50 mL, dosage of 0.5 g/L, temperature of 25 °C, oscillation time of 2 h, oscillation frequency of 100 r/min), with commercial coconut shell activated carbon as a reference. The results are shown in Table 1.

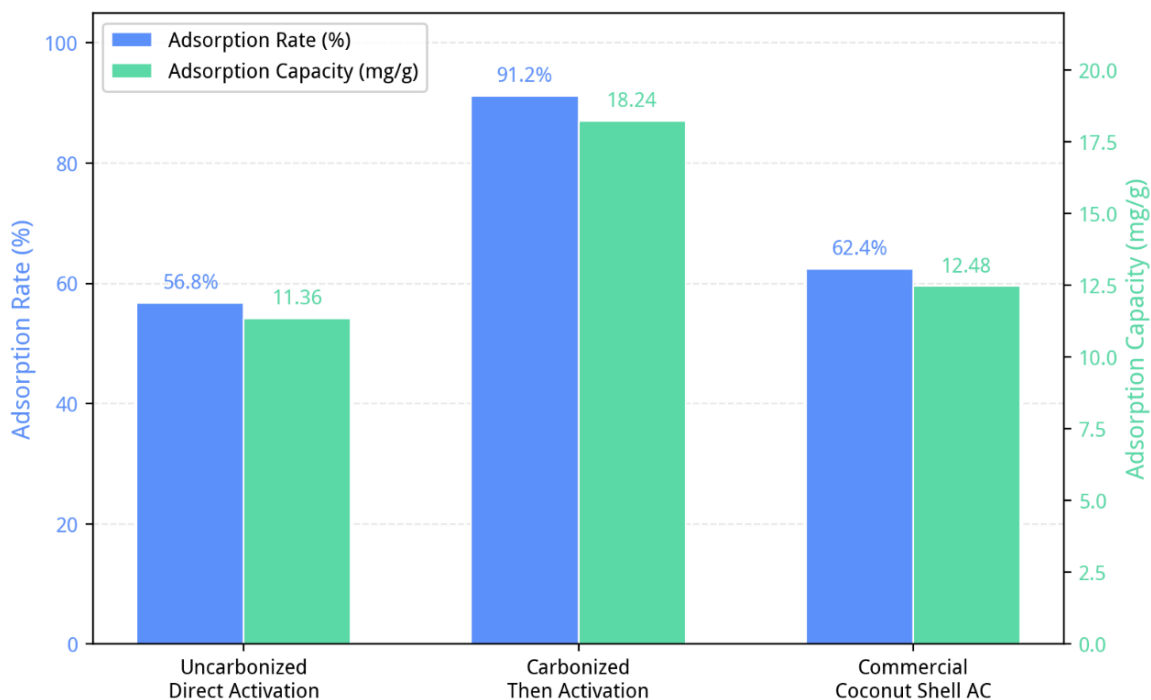


Figure 3: Comparison of Cu(II) Adsorption Performance of Activated Carbon with Different Precursor Pretreatment Methods

As shown in Figure 3, the adsorption rate and capacity of activated carbon prepared by carbonization followed by phosphoric acid activation are significantly higher than those of the sample activated directly without carbonization. The uncarbonized directly activated sample exhibits an adsorption rate of 56.8% and an adsorption capacity of 11.36 mg/g, while the carbonized then activated sample achieves an adsorption rate as high as 91.2% and an adsorption capacity of 18.24 mg/g, representing an improvement of 60.6%. This is because the pre-carbonization process pyrolyzes lignin, cellulose, and hemicellulose in the coconut shell raw material, releasing large amounts of volatile substances (CO , CO_2 , etc.) and preliminarily forming a carbon skeleton and primitive pore channels. This facilitates subsequent uniform penetration and etching of the phosphoric acid activating agent into the carbon matrix, thereby constructing a more developed hierarchical micro-mesoporous structure [9,14]. Additionally, the carbonization process increases the fixed carbon content and graphitization degree of the carbon material, enhancing the mechanical strength and chemical stability of the activated carbon and providing more effective sites for copper ion adsorption.

Commercial coconut shell activated carbon shows an adsorption rate of 62.4% and an adsorption capacity of 12.48 mg/g. The self-made carbonized material activated with phosphoric acid demonstrates clear advantages in adsorption performance, with an adsorption rate 28.8% higher. This is mainly attributed to the advantages of the phosphoric acid activation method in regulating pore structure and introducing oxygen-/phosphorus-containing functional groups. The prepared activated carbon has richer surface active sites and a more favorable pore size distribution, which is beneficial for the chemisorption of Cu^{2+} [8,11].

3.2.2 Effect of Solid-to-Liquid Ratio on Adsorption Performance

The solid-to-liquid ratio (g/mL) of phosphoric acid to carbonized material is a key process parameter affecting the pore structure and adsorption performance of activated carbon. Using uncarbonized coconut shell raw material and coconut shell carbonized material as precursors, three solid-to-liquid ratio levels (1:1, 1:2, 1:3) were set, and activated carbon was prepared at an activation temperature of 600 °C and an activation time of 1 h. The effect of the solid-to-liquid ratio on Cu^{2+} adsorption performance was investigated, and the results are shown in Table 2.

As shown in Figure 4, for the uncarbonized raw material direct activation system, as the solid-to-liquid ratio increases from 1:1 to 1:2, the adsorption rate increases from 45.2% to 56.8%, and the adsorption capacity increases from 9.04 mg/g to 11.36 mg/g. When the solid-to-liquid ratio further increases to 1:3, the adsorption rate instead decreases to 51.3%. For the carbonized material activation system, the adsorption rate is 68.5% at a ratio of 1:1, reaches a maximum of 91.2% at 1:2, and decreases to 84.7% at 1:3. Both systems exhibit the same trend: adsorption

performance first increases and then decreases with increasing solid-to-liquid ratio, indicating that 1:2 is the optimal solid-to-liquid ratio.

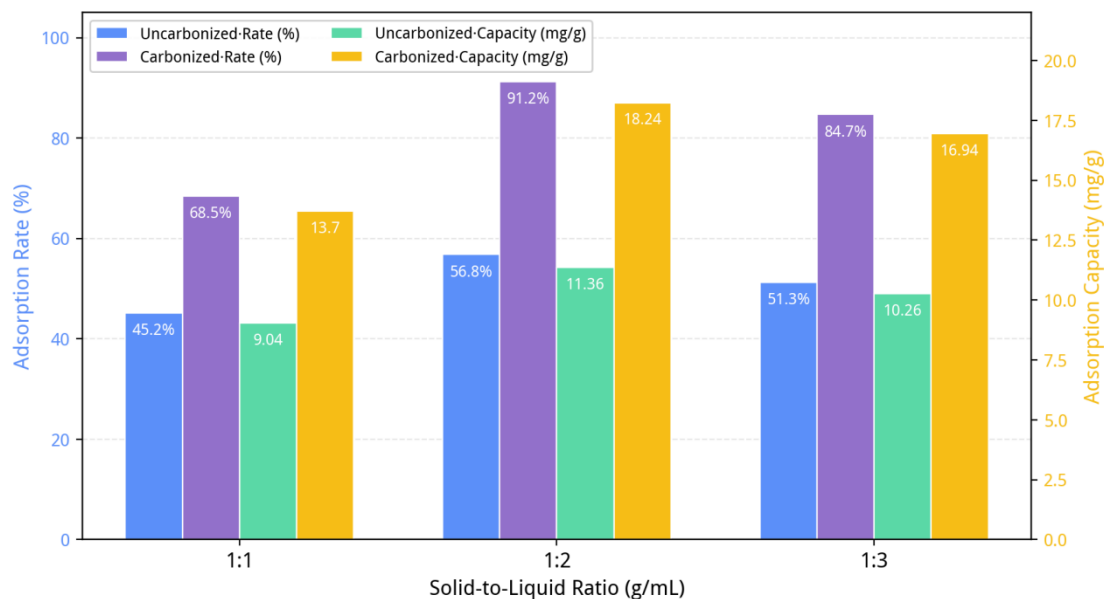


Figure 4: Cu(II) Adsorption Performance of Activated Carbon Prepared at Different Solid-to-Liquid Ratios

At a solid-to-liquid ratio of 1:1, the ratio is relatively low, and the phosphoric acid activating agent is insufficient to fully penetrate into the carbon matrix for etching and pore creation, resulting in underdeveloped pore structure, small specific surface area, and limited adsorption sites [8]. At a ratio of 1:2, the reaction between phosphoric acid and carbonaceous components is most complete; dehydrogenation, polycondensation, and oxidative etching synergistically construct abundant microporous and mesoporous structures while introducing an appropriate amount of phosphate ester functional groups, with both specific surface area and surface active sites reaching optimal levels [11,17]. At a ratio of 1:3, the ratio is excessively high; excess phosphoric acid causes over-etching, leading to micropore wall collapse, pore enlargement, and the conversion of some micropores into mesopores or even macropores, which conversely decreases the specific surface area. Meanwhile, excess phosphorus groups may block some pore channels, reducing the effective adsorption capacity of the activated carbon for Cu^{2+} [8,17]. This pattern is generally consistent with the literature-reported optimal impregnation ratio range (100%–150%) for phosphoric acid activation [17].

Comparing the two sets of data, the carbonized material activated samples outperform the uncarbonized directly activated samples at all solid-to-liquid ratio levels, further verifying the important role of the pre-carbonization step in improving phosphoric acid activation efficiency and activated carbon adsorption performance.

3.2.3 Effect of Adsorbent Dosage on Adsorption Performance

Using the carbonized material phosphoric acid-activated carbon prepared under optimal conditions (solid-to-liquid ratio of 1:2) as the adsorbent, the effect of adsorbent dosage (0.1–1.0 g/L) on Cu^{2+} adsorption performance was investigated (initial Cu^{2+} concentration of 100 mg/L, solution volume of 50 mL, temperature of 25 °C, oscillation time of 2 h). The results are shown in Table 1.

Table 1: Effect of Adsorbent Dosage on Cu(II) Adsorption Performance

Dosage (g/L)	Adsorbent Mass (g)	Adsorption Rate R (%)	Adsorption Capacity q_e (mg/g)
0.1	0.005	35.6	35.60
0.2	0.010	58.3	29.15
0.3	0.015	74.8	24.93
0.4	0.020	85.2	21.30
0.5	0.025	91.2	18.24
0.6	0.030	94.5	15.75
0.8	0.040	96.8	12.10
1.0	0.050	97.9	9.79

As shown in Table 1, as the activated carbon dosage increases from 0.1 g/L to 1.0 g/L, the copper ion adsorption rate continuously rises from 35.6% to 97.9%, while the unit mass adsorption capacity gradually decreases from 35.6 mg/g to 9.79 mg/g. The increase in adsorption rate is attributed to the fact that a higher dosage provides more adsorption sites and a larger effective specific surface area, enabling more Cu^{2+} to be captured [20,21]. However, the decrease in unit adsorption capacity occurs because, under fixed initial concentration and solution volume conditions, the total amount of Cu^{2+} available for adsorption in the solution is limited; an increased dosage leads to underutilization of adsorption sites per unit mass of adsorbent, resulting in a site surplus phenomenon [20].

When the dosage increases from 0.1 g/L to 0.5 g/L, the adsorption rate increases significantly (35.6%→91.2%), indicating that increasing the dosage within this range effectively improves removal efficiency. When the dosage exceeds 0.5 g/L, the increase in adsorption rate levels off (91.2% at 0.5 g/L, 97.9% at 1.0 g/L), indicating that adsorption is approaching equilibrium and the marginal benefit of further increasing the dosage diminishes [21,22]. Considering removal efficiency, adsorbent utilization, and treatment cost, 0.5 g/L was selected as the optimal dosage, at which the adsorption rate reaches 91.2% and the unit adsorption capacity is 18.24 mg/g, balancing treatment effectiveness and economy.

3.2.4 Comparison with Commercial Activated Carbon

Under identical experimental conditions (initial Cu^{2+} concentration of 100 mg/L, dosage of 0.5 g/L, temperature of 25 °C, oscillation time of 2 h), the adsorption performance of the activated carbon prepared under optimal conditions was compared with that of commercial activated carbon. The results show that the self-made activated carbon achieves a Cu^{2+} adsorption rate of 91.2% and an adsorption capacity of 18.24 mg/g, while the commercial activated carbon achieves an adsorption rate of 62.4% and an adsorption capacity of 12.48 mg/g. The adsorption rate and capacity of the self-made activated carbon are 46.2% and 46.2% higher than those of the commercial activated carbon, respectively.

The main reasons for the performance difference are: (1) The self-made activated carbon uses phosphoric acid chemical activation, which introduces abundant oxygen-containing functional groups ($-\text{OH}$, $-\text{COOH}$, $\text{P}-\text{O}-\text{C}$, etc.) on the carbon surface. These functional groups significantly enhance the chemisorption capacity for Cu^{2+} through complexation and ion exchange [7,8]. (2) The phosphoric acid activation process can precisely regulate the hierarchical micro-mesoporous structure, facilitating the diffusion and mass transfer of Cu^{2+} into the interior of the activated carbon [11]. (3) Commercial activated carbon mostly uses steam or CO_2 physical activation; although it has a high specific surface area, its surface functional groups are relatively scarce, limiting its chemisorption capacity for heavy metal ions [9,14]. The above results indicate that activated carbon prepared from coconut shell waste using the carbonization-phosphoric acid activation process exhibits good application potential and cost advantages in the field of copper-containing wastewater treatment.

4. CONCLUSIONS

In this study, coconut shell particles were used as raw material to prepare coconut shell activated carbon via the pre-carbonization-phosphoric acid activation method. Through SEM and FTIR characterization and static adsorption experiments, the effects of factors such as the phosphoric acid solid-to-liquid ratio and adsorbent dosage on the copper ion adsorption performance of activated carbon were systematically investigated, and a comparison with commercial activated carbon was conducted. The main conclusions are as follows:

1) The pre-carbonization step significantly promotes the adsorption performance of activated carbon. Activated carbon prepared by carbonization at 600 °C for 2 h followed by phosphoric acid activation exhibits much higher Cu^{2+} adsorption rate and capacity than the sample activated directly without carbonization, because pre-carbonization forms a preliminary carbon skeleton and pore channel structure that facilitates subsequent uniform penetration and efficient etching by the phosphoric acid activating agent.

2) The phosphoric acid solid-to-liquid ratio significantly affects the pore structure and adsorption performance of activated carbon. Adsorption performance first increases and then decreases with increasing solid-to-liquid ratio, with 1:2 being the optimal ratio. An excessively low ratio results in insufficient activating agent and inadequate pore creation; an excessively high ratio causes over-etching, leading to micropore collapse and a decrease in specific surface area.

3) The adsorbent dosage is positively correlated with the copper ion adsorption rate and negatively correlated with

the unit adsorption capacity. Considering removal efficiency and economy, the optimal dosage is 0.5 g/L, at which the adsorption rate reaches 91.2% and the unit adsorption capacity is 18.24 mg/g.

4) FTIR analysis shows that after adsorption, the peak positions of oxygen-containing functional groups such as hydroxyl (3400 cm^{-1}), carboxyl (1627 cm^{-1}), and C—O/P—O—C (1093 cm^{-1}) shift, and new Cu—O characteristic vibration peaks appear in the $400\text{--}700\text{ cm}^{-1}$ region, confirming that Cu^{2+} undergoes chemical complexation with surface oxygen-containing functional groups. The adsorption mechanism is dominated by chemisorption (surface complexation, ion exchange), supplemented by physical adsorption.

5) Under the optimal preparation conditions (carbonization at $600\text{ }^{\circ}\text{C}$ for 2 h + phosphoric acid activation at a solid-to-liquid ratio of 1:2, $600\text{ }^{\circ}\text{C}$ for 1 h) and adsorption conditions (dosage of 0.5 g/L, $25\text{ }^{\circ}\text{C}$, 2 h), the self-made coconut shell activated carbon achieves an adsorption rate of 91.2% for a 100 mg/L Cu^{2+} solution, significantly outperforming commercial coconut shell activated carbon and demonstrating good application prospects. This study provides a theoretical basis and technical reference for the resource utilization of agricultural and forestry wastes and the low-cost purification of copper-containing wastewater.

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