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PREPARATION OF STRAW-SLUDGE BASED ACTIVATED CARBON FOR THE TREATMENT OF PHOSPHORUS-CONTAINING WASTEWATER

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Abstract: In this study, rapeseed straw and dewatered sludge were used as raw materials and $ZnCl_2$ as an activator to prepare a straw-sludge based composite activated carbon via a mixed co-carbonization method. Single-factor and orthogonal experiments were conducted to investigate the effects of carbonization temperature, solid-liquid ratio, activation time, raw material ratio, and activator concentration on the iodine adsorption value of the composite activated carbon, aiming to optimize the preparation process. The results showed that the optimal preparation conditions for the composite activated carbon were: raw material ratio (sludge:straw) of 1:3, solid-liquid ratio of 1:3, activation time of 60 min, $ZnCl_2$ concentration of 3.5 mol/L, and carbonization temperature of 500 ℃. The composite activated carbon prepared under the optimal conditions was then utilized to adsorb PO_4^{3-} in water, and the effects of initial wastewater concentration, activated carbon dosage, and wastewater pH on the adsorption performance were investigated. The results demonstrated that under the conditions of an initial wastewater concentration of 40 mg/L, an activated carbon dosage of 40 g/L, and a pH of 7, the removal rate of PO_4^{3-} exceeded 93%, with an adsorption capacity of 1.17 mg/g.

Keywords: Straw; Sludge; Composite activated carbon; Adsorption performance; Phosphorus-containing wastewater

1 INTRODUCTION

With the acceleration of urbanization and the large-scale development of agricultural production, the discharge of solid wastes, such as excess sludge from municipal wastewater treatment plants and straw from agricultural production, has been increasing year by year. According to statistics, China's annual sludge production (with a moisture content of 80%) exceeds 60 million tons, and the annual straw output reaches over 700 million tons. If improperly disposed of, these solid wastes will not only occupy vast land resources but also potentially cause environmental issues such as soil and water pollution through leakage and volatilization, threatening ecological security and human health. Therefore, achieving the reduction, harmlessness, and resource utilization of sludge and agricultural and forestry wastes has become a significant topic in the current field of environmental protection [1].

Phosphorus-containing wastewater is one of the main pollution sources causing water eutrophication. Excessive phosphorus leads to massive algal proliferation in water bodies, disrupts the aquatic ecological balance, and affects the normal utilization of water resources. Currently, the primary treatment methods for phosphorus-containing wastewater include chemical precipitation, biological treatment, and adsorption. Among them, the adsorption method has attracted considerable attention in treating low-concentration phosphorus-containing wastewater due to its advantages of simple operation, low cost, and high adsorption efficiency. As a traditional adsorbent, activated carbon features a large specific surface area and a well-developed pore structure. However, activated carbon prepared from a single raw material often suffers from limited adsorption performance and relatively high costs.

Sludge contains abundant organic matter and minerals, which can be converted into sludge-based activated carbon with adsorption properties after proper treatment. Meanwhile, straw is rich in carbon elements and natural fibers, possessing hydrophilicity and a porous structure, making it a high-quality auxiliary raw material for activated carbon preparation. Currently, the preparation of sludge-based activated carbon typically adopts a carbonization-activation process, with common activators including $ZnCl_2$, KOH, and H_3PO_4. Numerous studies have shown that sludge-based activated carbon possesses a certain adsorption capacity for pollutants such as heavy metals, organic matter, and phosphorus in water. For instance, Shao Ruihua (2016) prepared sludge-derived activated carbon using sludge as the raw material [2], and the study found that its adsorption capacity for phosphorus-containing wastewater could reach a certain level. However, single sludge-based activated carbon still faces issues such as a small specific surface area and an underdeveloped pore structure, which limit the further enhancement of its adsorption performance. To improve this situation, researchers have attempted to enhance the adsorption performance of sludge-based activated carbon by doping it with other materials[3].

Preparing activated carbon through the co-composite of straw and sludge not only solves the disposal dilemmas of both wastes but also enhances the adsorption performance of the activated carbon through synergistic effects, reduces preparation costs, and realizes the environmental concept of "treating waste with waste". Based on this, this study investigates the adsorption performance enhancement of sludge-based activated carbon by doping with crop straw, aiming to optimize the preparation process of the composite activated carbon, clarify its adsorption characteristics and mechanisms for phosphorus in water, and provide technical support and theoretical basis for the efficient treatment of phosphorus-containing wastewater.

2 MATERIALS AND METHODS

2.1 Main Reagents and Instruments

Instruments:101-2DB electric thermostatic drying oven (Taiste Instrument Co., Ltd.); MF-20 high-speed multi-function crusher (Wuyi Haina Electric Co., Ltd.); SX2-4-13 high-temperature box furnace at 1300 °C (Hubei Yingshan Jianli Electric Furnace Manufacturing Co., Ltd.); ME104E analytical balance (Shanghai Zanwei Weighing Apparatus Co., Ltd.); 1000 μL and 5000 μL pipettes (Dragonlab Instruments Co., Ltd.); HZQ-F160 vertical air thermostatic oscillating incubator (Changzhou Nuoji Instrument Co., Ltd.); HZQ-F100 full-temperature oscillating incubator (Taichang Haocheng Experimental Instrument Manufacturing Co., Ltd.); UV-2600 UV spectrophotometer (Shanghai Angla Instrument Co., Ltd.).

Reagents:Concentrated hydrochloric acid, iodine, potassium iodide, sodium thiosulfate ($Na_2S_2O_3\cdot 5H_2O$), soluble starch, potassium iodate, anhydrous sodium carbonate, potassium dihydrogen phosphate, sulfuric acid solution, ascorbic acid, ammonium molybdate, sodium hydroxide, potassium antimony tartrate ($KSbOC_4H_4O_6\cdot 1/2H_2O$).

2.2 Experimental Procedures and Methods

2.2.1 Preparation method of straw-sludge based composite activated carbon

(1) After air-drying, the straw and sludge were separately placed in a 105 ℃ oven and dried to constant weight. After being taken out and cooled, they were ground and sieved through a 200-mesh sieve. Straw powder and sludge powder were weighed according to a certain ratio, mixed evenly with $ZnCl_2$ solution as the activator, and activated at room temperature for a specific time. Subsequently, the mixture was placed in an electric thermostatic drying oven and dried at 105 ℃ for 24 h. Under the preset carbonization temperature and time conditions, the activated and dried sample was placed in a muffle furnace for carbonization. After carbonization and cooling, the sample was first soaked with 3 mol/L HCl for 20 min, then washed with deionized water to neutral, and dried again in a 105 ℃ oven for 24 h. Finally, it was ground to a particle size of 20-40 mesh to obtain the straw-sludge based composite activated carbon, which was stored in a desiccator for future use.

(2) Single-factor experiments were designed using the iodine adsorption value as the evaluation index to investigate the effects of five factors on the performance of the prepared straw-sludge based composite activated carbon: temperature (400 ℃, 450 ℃, 500 ℃, 550 ℃, 600 ℃), solid-liquid ratio (2:3, 1:2, 2:5, 1:3, 1:4), activation time (0.5 h, 1 h, 1.5 h, 2 h, 2.5 h), raw material ratio (2:1, 1:1, 1:2, 1:3, 1:4), and activator concentration (1 mol/L, 2 mol/L, 2.5 mol/L, 3 mol/L, 3.5 mol/L). Furthermore, a five-factor and five-level orthogonal experiment was designed for further analysis to determine the significance of each factor, thereby identifying the optimal combination of preparation conditions [4].

2.2.2 Index determination of straw-sludge based composite activated carbon

Determination of the iodine adsorption value of the composite activated carbon:"Test methods of wooden activated carbon - Determination of iodine number"(GB/T 12496.8-2015).

The iodine adsorption value A is calculated according to Equation (1):

$$A=\frac{X}{M}\cdot D \tag{1}$$

2.2.3 Adsorption performance of straw-sludge based composite activated carbon for PO_4^{3-} in water

Adsorption studies were conducted on simulated PO_4^{3-}-containing wastewater using the straw-sludge based composite activated carbon prepared under the optimal conditions. The experimental parameters were set as follows: a wastewater volume of 50 mL and an oscillation speed of 120 r/min for 4 h. The composite activated carbon dosage (0.5 g, 1.0 g, 1.5 g, 2.0 g, 2.5 g), initial concentration of the phosphorus-containing wastewater, and solution pH were selected as the influencing factors. When a single factor was varied while keeping others constant, the adsorption capacity of the composite activated carbon for PO_4^{3-} under different conditions of this factor was determined, and the corresponding removal rate was subsequently calculated.

The calculation of the removal rate of PO_4^{3-} by the straw-sludge based composite activated carbon is shown in Equation (2-2), and the calculation of the adsorption capacity is shown in Equation (2).

$$\eta=(C_O-C_e)/Co*100\% \tag{2}$$

$$Q_e=(Co-Ce)*v/m \tag{3}$$

In the formula:

C_o — initial concentration of PO_4^{3-} in the wastewater sample, mg/L;

C_e — concentration of PO_4^{3-} in the solution at adsorption equilibrium, mg/L;

Q_e — adsorption capacity of the activated carbon, mg/g

m — dosage of the activated carbon, g

V — volume of the PO_4^{3-}-containing wastewater, L.

3 RESULTS AND DISCUSSION

3.1 Single-Factor Experiments of Straw-Sludge Based Composite Activated Carbon

3.1.1 Effect of temperature on the preparation of composite activated carbon

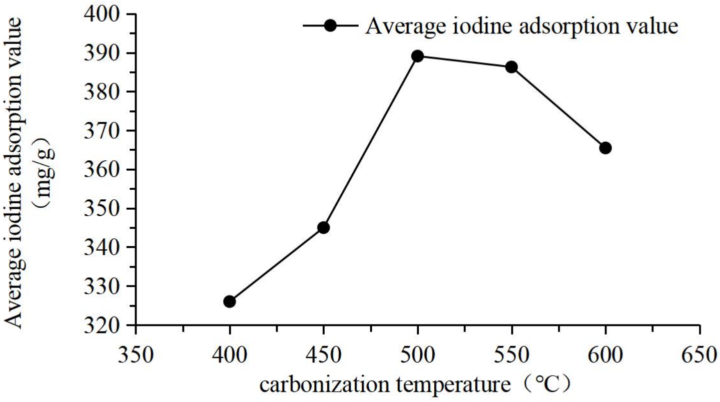

Figure 1 The Influence of Temperature on the Adsorption Effect of Iodine

As shown in Figure 1, with the increase in temperature, the average iodine adsorption value initially increased and then decreased, reaching a maximum of 389.09 mg/g at a carbonization temperature of 500 ℃. When the temperature was below 500 ℃, the iodine adsorption value exhibited a significant upward trend with increasing temperature; at 500 ℃, the iodine adsorption value reached its peak. As the temperature continued to rise, the iodine adsorption value was 386.29 mg/g at 550 ℃, showing little difference from that at 500 ℃. However, when the temperature exceeded 550 ℃ and further increased to 600 ℃, the iodine adsorption value decreased significantly. This phenomenon can be explained as follows: at lower temperatures, the degree of pyrolysis is relatively low, and the raw materials are not fully activated. The pore structure of the resulting activated carbon is underdeveloped, leading to a poor adsorption capacity. As the temperature rises, the degree of pyrolysis gradually increases, and the pore structure of the activated carbon becomes more developed, thereby greatly enhancing the adsorption performance. At excessively high temperatures, the carbon in the straw and sludge is burned off, a large amount of zinc chloride is lost, and the pore structure collapses, resulting in a decrease in the iodine adsorption value, that is, a decline in the adsorption performance of the activated carbon. Based on this analysis, the optimal carbonization temperature range is 500 ℃-550 ℃, and 500 ℃ is selected as the optimal carbonization temperature [3].

3.1.2 Effect of solid-liquid ratio on the preparation of composite activated carbon

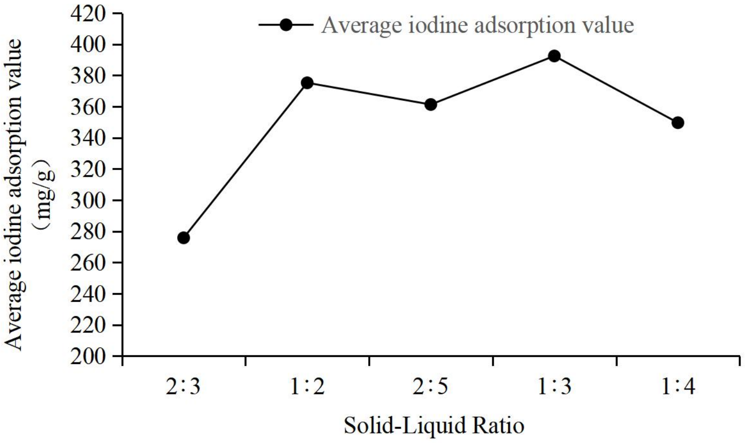

Figure 2 Effect of Solid-Liquid Ratio on Iodine Adsorption Performance

As shown in Figure 2, within the solid-liquid ratio (straw-sludge powder g : $ZnCl_2$ solution volume mL) range of 2:3 to 2:5, the average iodine adsorption value of the composite activated carbon gradually decreased with the increasing proportion of the $ZnCl_2$ activator solution. When the solid-liquid ratio was 2:5, the average iodine adsorption value reached its minimum of 349.65 mg/g. Between the solid-liquid ratios of 2:5 and 1:3, the average iodine adsorption value increased, reaching a maximum of 392.45 mg/g at 1:3. Subsequently, the average iodine adsorption value decreased again as the proportion of the $ZnCl_2$ activator solution continued to increase. This is because $ZnCl_2$ plays roles in pore-forming and inhibiting tar generation; it etches the organic matter in the raw materials, leading to the escape of water vapor and small molecular hydrocarbons, and consequently forming carbon microcrystallites with a highly porous structure. As the dosage of the $ZnCl_2$ activator increases, more carbon microcrystallites are generated in the composite activated carbon, resulting in a more developed pore structure and a larger specific surface area of the carbon, which yields a better adsorption performance. However, with an excessive addition of the $ZnCl_2$ activator, the surplus activator remains in the pores and blocks the pore structure, thereby impairing the adsorption performance. Based on this analysis, a solid-liquid ratio of 1:3 is optimal for adsorption.

3.1.3 Effect of activation time on the preparation of composite activated carbon

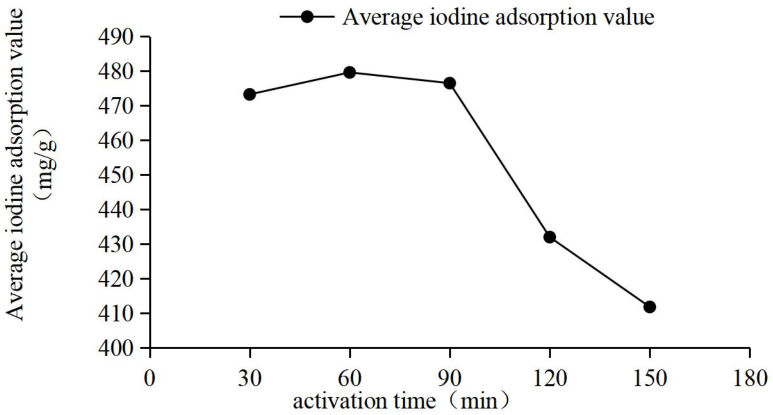

Figure 3 The Influence of Activation Time on the Adsorption Effect of Iodine

As shown in Figure 3, within the activation time range of 30 min to 150 min, the average iodine adsorption value of the composite activated carbon exhibited a trend of initially increasing and then decreasing. When the activation time was less than 60 min, the iodine adsorption value of the activated carbon gradually increased with the prolongation of the activation time, and its performance gradually improved; at an activation time of 60 min, the average iodine adsorption value of the composite activated carbon reached a maximum of 479.63 mg/g. When the activation time exceeded 60 min, the iodine adsorption value of the activated carbon declined, and the decrease became significant after exceeding 90 min. This phenomenon can be explained as follows: with the prolongation of the activation time, the activation reaction between the composite activated carbon and zinc chloride becomes progressively more complete, leading to a more developed pore structure and a corresponding increase in adsorption performance. However, once the activation time reaches a certain threshold, the pores of the composite activated carbon continue to expand, turning from micropores into mesopores and macropores, which results in a decrease in the specific surface area. Moreover, the massive loss of organic matter also leads to a decline in the adsorption performance of the composite activated carbon. Based on this analysis, the optimal adsorption effect is achieved at an activation time of 60 min.

3.1.4 Effect of raw material ratio on the preparation of composite activated carbon

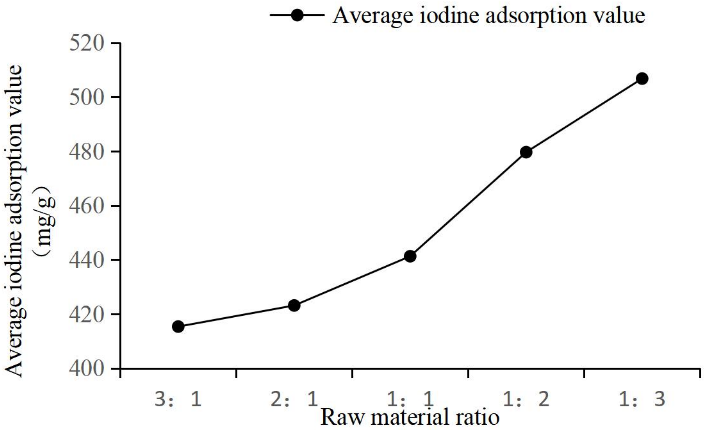

Figure 4 Effect of Raw Material Ratio on Iodine Adsorption

As shown in Figure 4, within the raw material ratio range of 3:1 to 1:3, the average iodine adsorption value of the straw-sludge based composite activated carbon increased with the increasing proportion of straw. At a raw material ratio of 1:3, the average iodine adsorption value reached a maximum of 506.75 mg/g. This is because a higher carbon element content leads to a higher iodine adsorption value of the adsorbent. Although dewatered sludge contains a certain amount of carbon, its carbon content is relatively low compared to that of rapeseed straw. Rapeseed straw, on the other hand, has a high carbon content, and its natural fibers possess excellent hydrophilicity and a rich, diverse porous structure. Therefore, the inherent characteristics of straw are beneficial for enhancing the adsorption performance of activated carbon and compensate for the drawback of low carbon content in the sludge [5]. Since this study aims not only to explore the preparation of activated carbon adsorbents, but also to achieve effective resource utilization of the sludge, the addition of rapeseed straw is intended to increase the carbon content of the sludge-based activated carbon. Therefore, considering all factors, a raw material ratio range of 3:1 to 1:3 was selected, and the adsorption effect is optimal at a ratio of 1:3.

3.1.5 Effect of activator concentration on the preparation of composite activated carbon

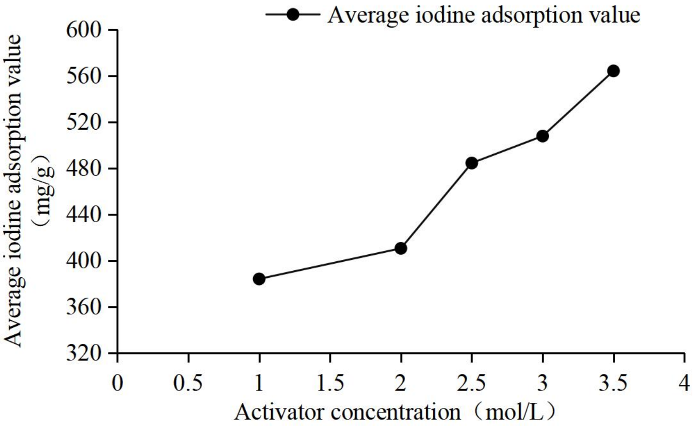

Figure 5 The Influence of Activator Concentration on the Adsorption Effect of Iodine

As shown in Figure 5, within the $ZnCl_2$ activator concentration range from 1 mol/L to 3.5 mol/L, the average iodine adsorption value of the straw-sludge based composite activated carbon gradually increased with the increase in activator concentration. When the $ZnCl_2$ activator concentration was 3.5 mol/L, the average iodine adsorption value reached its maximum of 564.12 mg/g. This is because, with the increase in $ZnCl_2$ activator concentration, the pore structure of the activated carbon becomes more developed and the pore distribution becomes broader, thereby enhancing the adsorption performance and facilitating the progress of adsorption. Based on this analysis, the optimal adsorption effect is achieved at a $ZnCl_2$ activator concentration of 3.5 mol/L [6].

3.2 Orthogonal Experiment of Straw-Sludge Based Composite Activated Carbon

3.2.1 Orthogonal factor and level table

Based on the above single-factor experiment results, five factors—carbonization temperature, activator concentration, raw material ratio, solid-liquid ratio, and activation time—were selected with representative values, and the $L_{25}(5^5)$ orthogonal array was adopted. The experimental design of the five-factor and five-level orthogonal table for the preparation of straw-sludge based activated carbon is shown in Table 1.

Table 1 Orthogonal Experimental Design Table

Factor Level	Carbonization temperature (°C)	Activator concentration (mg/L)	Raw material ratio	Solid-liquid ratio	Activation time (h)
1	400	1	3: 1	2: 3	0.5
2	450	2	2: 1	1: 2	1
3	500	2.5	1: 1	2: 5	1.5
4	550	3	1: 2	1: 3	2
5	600	3.5	1: 3	1: 4	2.5

3.2.2 Orthogonal experimental results

Table 2 Orthogonal Experimental Design Table

Number	Carbonization temperature (°C)	Activator concentration (mg/L)	Raw material ratio	Solid-liquid ratio	Activation time (h)	Iodine adsorption value mg/g
1	400	1	3:1	2:3	0.5	113.42
2	400	2	2:1	1:2	1	219.04
3	400	2.5	1:1	2:5	1.5	306.13
4	400	3	1:2	1:3	2	365.95
5	400	3.5	1:3	1:4	2.5	343.74
6	450	1	2:1	2:5	2	163.46
7	450	2	1:1	1:3	2.5	241.49

8	450	2.5	1:2	1:4	0.5	189.78
9	450	3	1:3	2:3	1	357.27
10	450	3.5	3:1	1:2	1.5	116.49
11	500	1	1:1	1:4	1	174.59
12	500	2	1:2	2:3	1.5	192.66
13	500	2.5	1:3	1:2	2	220.70
14	500	3	3:1	2:5	2.5	141.37
15	500	3.5	2:1	1:3	0.5	135.21
16	550	1	1:2	1:2	2.5	322.15
17	550	2	1:3	2:5	0.5	405.47
18	550	2.5	3:1	1:3	1	163.93
19	550	3	2:1	1:4	1.5	188.18
20	550	3.5	1:1	2:3	2	318.45
21	600	1	1:3	1:3	1.5	493.78
22	600	2	3:1	1:4	2	214.74
23	600	2.5	2:1	2:3	2.5	253.43
24	600	3	1:1	1:2	0.5	300.91
25	600	3.5	1:2	2:5	1	428.16
$\overline{\text{I}}$	269.66	253.48	149.99	247.05	228.96	
$\overline{\text{II}}$	213.70	254.68	191.86	235.86	268.60	
$\overline{\text{III}}$	172.91	226.79	268.31	288.92	259.45	
$\overline{\text{IV}}$	279.64	270.74	299.74	280.07	256.66	
$\overline{\text{V}}$	338.20	268.41	462.95	222.21	260.44	
R	165.30	43.94	312.96	66.71	39.64	

The results of the five-factor and five-level orthogonal experiment for the preparation of straw-sludge based activated carbon are shown in Table 2. Taking the range value R as the evaluation index, there is a positive correlation between the R value and the influence degree of each factor. Therefore, the influence degree of each factor can be determined by comparing the magnitude of the range value R.

As shown in Table 2, the ranges of the average iodine adsorption values for the five factors—carbonization temperature, activator concentration, raw material ratio, solid-liquid ratio, and activation time—are 165.30 mg/g, 43.94 mg/g, 312.96 mg/g, 66.71 mg/g, and 39.64 mg/g, respectively. Therefore, the sequence of their influence degrees on the preparation of the straw-sludge based composite activated carbon is as follows: raw material ratio > carbonization temperature > solid-liquid ratio > activator concentration > activation time.

3.3 The Adsorption Performance of Straw-Sludge Based Composite Activated Carbon for PO_4^{3-} in Water

3.3.1 Effect of straw-sludge based composite activated carbon dosage on phosphorus adsorption

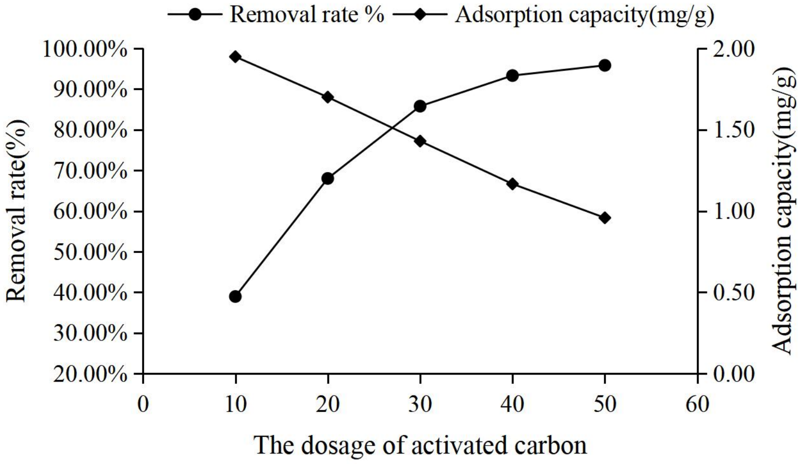

Figure 6 Effect of Dosage on Orthophosphate Phosphorus Removal Rate and Activated Carbon Adsorption Capacity

As shown in Figure 6, with the increase in the dosage of straw-sludge based composite 、activated carbon, the removal rate of orthophosphate in wastewater gradually increases. When the dosage is 30 g/L, the removal rate reaches 85.78%; when the dosage increases to 40 g/L and 50 g/L, the removal rates are both above 93%, achieving a good removal effect. However, as the dosage increases, the adsorption capacity of the activated carbon shows an inverse growth. At a dosage of 10 g/L, the adsorption capacity reaches 1.95 mg/L; at dosages of 40 g/L and 50 g/L, the adsorption capacities are 1.17 mg/L and 0.96 mg/L, respectively. The increase in the removal rate of orthophosphate is mainly due to the increase of orthophosphate adsorption sites per unit volume of solution. The main reason for the decrease in phosphorus adsorption capacity is the interaction between electrostatic adsorption sites or the interaction between adsorption sites, which reduces the effective specific surface area of the sludge-based activated carbon and leads to an extended diffusion path [7-8]. Based on the comprehensive analysis above, taking both the removal rate and adsorption capacity of the activated carbon into consideration, a dosage of 40 g/L is considered the optimal dosage.

3.3.2 Effect of initial solution concentration on phosphorus adsorption

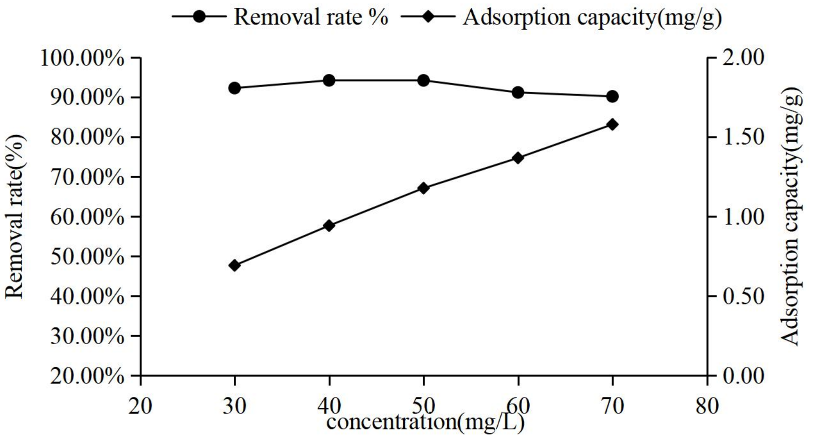

Figure 7 Effect of Initial Solution Concentration on orthophosphate Phosphorus Removal Rate and Activated Carbon Adsorption Capacity

As shown in Figure 7, under the condition of a dosage of 40 mg/L and phosphate concentrations ranging from 30 to 70 mg/L, the removal rates of orthophosphate all exceed 90%. Among them, when the solution concentration is 40 mg/L, the removal rate reaches its maximum at 94.19%. In this experiment, the adsorption capacity gradually increases with the increase of the initial solution concentration; when treating an initial solution concentration of 70 mg/L, the adsorption capacity of the activated carbon is 1.58 mg/g. The experiment demonstrates that at a dosage of 40 mg/L, a high removal rate can be achieved for high-concentration phosphorus-containing wastewater, and a good adsorption effect can be obtained.

3.3.3 Effect of initial solution pH on phosphorus adsorption

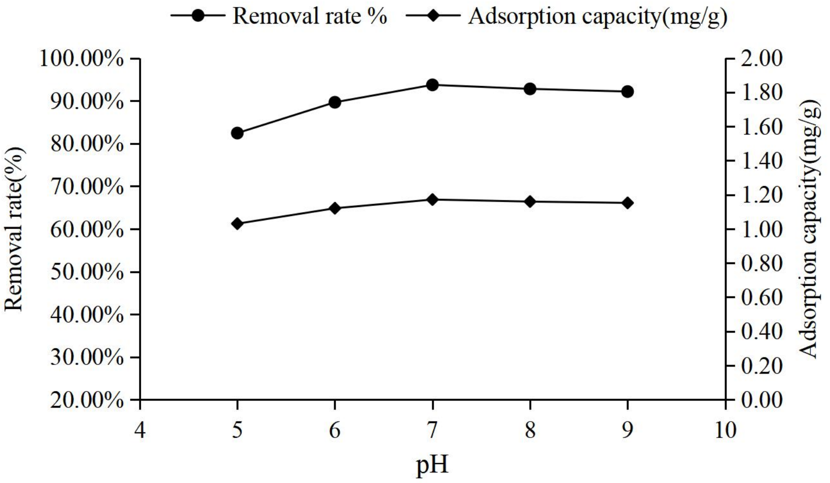

Figure 8 Effect of Initial Solution pH on Orthophosphate Phosphorus Removal Rate and Activated Carbon Adsorption Capacity

As shown in Figure 8, within the pH range of 5 to 7, the phosphorus removal rate by the composite activated carbon gradually increases, while within the pH range of 7 to 9, it gradually decreases. The overall removal rate is above 87%, with the phosphorus removal rate reaching a maximum of 93.73% and the adsorption capacity reaching 1.17 mg/g at pH 7. Under acidic conditions, the surface of the activated carbon is positively charged, which effectively enhances its attraction to PO_4^{3-} [9]. As the pH gradually approaches neutrality, the concentrations of HPO_4^{2-} and PO_4^{3-} in the system increase, which enhances the opportunities for ion exchange between the phosphate and the surface of the activated carbon. With the further increase of pH, the increase in OH^- leads to competitive adsorption between OH^- and phosphate, resulting in a gradual decrease in the phosphorus removal rate by the activated carbon [10]. Therefore, it can be concluded that under neutral conditions, the phosphorus removal rate and adsorption capacity of the activated carbon are optimal.

4 CONCLUSION

In this study, dewatered sludge and rapeseed straw were used as raw materials to prepare a composite activated carbon via $ZnCl_2$ activation and mixed co-carbonization. The preparation process was systematically optimized, and its adsorption performance for PO_4^{3-} in water was investigated. The main conclusions are drawn as follows:

(1) The results of single-factor experiments and orthogonal experiments for the preparation of the composite activated carbon show that the raw material ratio has the greatest impact on the iodine adsorption value of the activated carbon, followed by carbonization temperature, solid-liquid ratio, and activator concentration, while the activation time has a relatively minor impact. The optimal preparation conditions are: raw material ratio (sludge : straw) of 1:3, solid-liquid ratio of 1:3, activation time of 60 min, activator $ZnCl_2$ concentration of 3.5 mol/L, and carbonization temperature of 500 ℃.

(2) The adsorption experiment results of the straw-sludge based composite activated carbon for PO_4^{3-} in water demonstrate that when the dosage is 40 g/L, the initial wastewater concentration is 40 mg/L, and the pH is 7 (neutral), the PO_4^{3-} removal rate reaches over 93% with an adsorption capacity of 1.17 mg/g; the activated carbon still maintains a high removal rate (over 90%) and an adsorption capacity of 1.58 mg/g for high-concentration phosphorus-containing wastewater (70 mg/L), indicating its good adaptability in the treatment of phosphorus-containing wastewater at different concentrations.

(3) This study achieved the enhancement of the adsorption performance of sludge-based activated carbon through straw doping, which not only solves the resource utilization problem of sludge and straw, but also provides a low-cost and efficient adsorbent material for the treatment of phosphorus-containing wastewater.

COMPETING INTERESTS

The authors have no relevant financial or non-financial interests to disclose.

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