

Article

A Preliminary Investigation on the Adsorption of Cu^{2+} by Sawdust/Foamed Geopolymer Composites

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Abstract

Sawdust is receiving increasing attention as a promising green adsorbent. However, due to its powder nature, it is difficult to recover after adsorbing heavy metals and may even cause secondary pollution. To solve this problem, a novel sawdust/foamed geopolymer (SFG) adsorbent was prepared by using sawdust as a raw material, geopolymer as a binder, and hydrogen peroxide as a foaming agent. This study discussed the effect of SFG dosage, solution temperature, solution pH, contact time, and initial Cu^{2+} solution concentration on the adsorption capacity and removal rate. The results showed that a desirable SFG adsorbent with the SFG dosage of 0.5 g, temperature of 25 °C, pH of 5, contact time of 720 min, and initial Cu^{2+} solution concentrations of 90 mg/L is recommended, of which the adsorption capacity is 31.5 mg/g with the removal rate being 92.76%. In addition, the adsorption performance of the SFG adsorbent is superior to that of pure sawdust and similar to that of the foamed geopolymer adsorbent, and it has the characteristics of higher strength, lower cost, and more environmental friendliness. This study indicated that the SFG adsorbents are feasible as adsorbents; meanwhile, this work can provide a scientific reference for the development of new bio-composite adsorbent materials, especially in the field of the treatment of heavy metal ions in wastewater.

Keywords: waste sawdust; foamed geopolymer; heavy metal Cu^{2+} ; adsorption capacity; removal rate



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1. Introduction

In recent years, with the improvement of people's living standards, the amount of rural living waste has also increased, including not only rotten straw, vegetable leaves, and fruit peels but also plastic bags, waste batteries, pesticide bottles, etc. However, most rural areas have not established a garbage collection and disposal system, especially in villages located in mountainous areas, so these wastes cannot be collected in a timely manner and effectively treated. If they are only differentiated and decomposed under natural conditions, a large amount of acidic and alkaline organic pollutants and heavy metals will be produced, resulting in serious pollution of surface water and groundwater. At the same time, due to dispersed rural living and the lack of centralized sewage treatment systems, a

large amount of domestic sewage is directly discharged into fields, rivers, or lakes. This phenomenon will seriously affect the promotion of rural revitalization and contradict the concept of green and low-carbon development. Therefore, how to treat rural wastewater, especially heavy metal ions that can cause serious degradation to the environment and health problems to humans, is a question worth exploring.

According to the statistics, the percentage of forest cover is 22.96% [1], and the timber production was 121.93 million cubic meters in 2022 [2]. It is well known that sawdust is a waste by-product from wood processing in construction work and furniture, and its traditional disposal method is centralized burning [3]. With the increased interest in environmental protection and energy saving, sawdust powder has many potential applications in various fields. Due to the fact that the main components of sawdust are lignin and cellulose, its surface contains a large number of active groups such as hydroxyl and carboxyl groups [4]. Furthermore, it has more micropores and a larger specific surface area in its structure. Therefore, the reuse of sawdust in the preparation of adsorbent materials is becoming increasingly widespread, especially in the adsorption of heavy metal ions [5,6].

However, it is worth noting that raw sawdust has a limited adsorption capacity, and its powdery state makes it difficult to separate from water, which extremely limits its practical application. To enhance the adsorption capacity of raw sawdust, it needs to be modified or combined with other high-strength adsorbents. Faisal M et al. [7] evaluated the performance of removing Cd(II) ions from wastewater using raw sawdust and modified sawdust with sulfuric acid as adsorbents. The experimental results showed that the Cd(II) ions removal percentage by modified sawdust (removal rate: 95.51%) is better than that of raw sawdust (removal rate: 82.22%). Samal et al. [8] prepared a new sawdust composite by a green technique comprised of mild phosphoric acid activation of commercial sawdust. The results showed that the sawdust composite has an excellent Cr(VI) adsorption capacity of 193.913 mg/g. In addition, it has good regeneration ability, which is of great significance for the sustainable development of the water treatment field. Mishra et al. [9] studied the effect of adding highly conductive polymers to sawdust on lead (Pb(II)) ions adsorption capacity. The results showed that the highest lead uptake capacity is 218 mg/g (at 318 K), and the modified sawdust with porous and one-dimensional flake-like structure is the main reason for its improved adsorption capacity. Hajam et al. [10] investigated the effects of different influencing factors (pH, contact time, initial metal concentration, mass of adsorbent, and temperature of the medium) on the Pb²⁺ ions adsorption performance of raw and chemically activated sawdust. The results showed that the removal rates of Pb²⁺ ions by raw and activated sawdust reached the highest values of 94% and 99%, respectively, under the optimum conditions (pH = 6, the mass of adsorbent = 0.875 g, contact time = 90 min (raw sawdust)/47.5 min (activated sawdust), and metallic ion concentration = 275 mg/L).

Geopolymer is an inorganic polymer material that exhibits less environmental footprint (e.g., less CO₂ emission, lower energy use, etc.) and excellent properties (e.g., low thermal conductivity, high mechanical, and good chemical resistance) [11–13]. Owing to its unique three-dimensional structural network, geopolymer also exhibits excellent heavy metal adsorption capacity [14–16]. However, to obtain a high surface area that can greatly enhance the extent of adsorption, the most common method is to prepare geopolymer adsorbents in powder form. However, this method also brings additional problems, including difficulty in collecting after exhaustion, creating secondary contamination, increased complexity, and operating costs of post-separation. Therefore, to solve these problems, the foamed geopolymer with a higher surface area is an attractive choice as a porous bulk-type geopolymer adsorbent [17,18]. Many researchers have carried out the adsorption of heavy

metals by foam geopolymers in the field of wastewater treatment. Liang et al. [19] developed a porous geopolymer adsorbent (PGA) with high strength and high Cu^{2+} adsorption ability by adding nano-silica. The results showed that when the dosage of nano-silica is 2%, the PGA can achieve a compressive strength of 25.3 MPa and a Cu^{2+} removal rate of 78.4%. Moreover, PGA can still maintain its integrity underwater after adsorbing heavy metals, which provides great convenience for practical engineering applications. Liu et al. [20] developed a novel porous phosphoric acid-activated geopolymer foam (PAG) with an open cell structure. The experimental results showed that at $\text{pH} = 7$, the maximum removal efficiencies of Pb^{2+} , Ni^{2+} , and Cd^{2+} are 11.99 mg/g, 6.16 mg/g, and 2.81 mg/g, respectively, which has a high potential application in wastewater treatment. Tan et al. [21] proved that foaming can overcome the primary limitation of the low specific surface area of bulk-type adsorbent, and the Cu^{2+} removal capacity of foamed geopolymer spheres is six times that of non-foamed geopolymer spheres. Ji et al. [22] produced a composite waste-based porous open-cell geopolymer (POG) from industrial and municipal wastes. The results showed that POG has a significant adsorption advantage for heavy metal cations, and the removal rates of Cd^{2+} , Cu^{2+} , and Pb^{2+} by POG are as high as 99.9%, 95.49%, and 98.52%, respectively.

As mentioned before, scholars have done some research on the adsorption performance of sawdust and foam geopolymer for heavy metals. However, these studies focused on single sawdust or foam geopolymer as adsorbents. If sawdust can be used as a substitute for some of the raw materials required for the preparation of foamed geopolymer to create a new type of adsorbent, while also improving the overall heavy metal adsorption capacity, this will not only reduce the cost but also have an important guiding significance for the development of new environmentally friendly adsorbent. According to the author's investigation, the adsorption properties of this new material using sawdust and foam geopolymer coupling as adsorbents have not yet been reported.

From the above perspectives, the purpose of this study is to evaluate the heavy metal adsorption capacity of sawdust/foamed geopolymer (SFG) adsorbents. Meanwhile, this experiment chose heavy metal Cu^{2+} ions as the adsorption target, and the removal rate and adsorption capacity were used as performance evaluation indicators. To explore the main factors affecting the adsorption performance of SFG adsorbents, the microstructure characteristics of adsorbents were observed, and the effect of SFG dosage, solution temperature, solution pH, contact time, and initial Cu^{2+} solution concentrations on the adsorption process of Cu^{2+} was researched.

The content of this paper is structured as follows: (1) background; (2) the exhibition of experimental raw materials, sample preparation, and characterization methods; (3) discussion of the effect of SFG dosage, adsorption temperature, solution pH, and initial Cu^{2+} concentration on the removal rate and adsorption capacity and analysis of EDS and XRD test results; (4) comparison and analysis of adsorption properties of sawdust, foamed geopolymer, and SFG; and (5) conclusions.

2. Materials and Methods

2.1. Materials

2.1.1. Raw Materials

During the sawdust/foamed geopolymer adsorbent preparation process, metakaolin and waste sawdust were used as the main materials. Sodium hydroxide, sodium silicate solution, hydrogen peroxide, soap powder, and distilled water were used as the main reagents. The detailed parameters of raw materials were introduced as follows:

1. The metakaolin (1250 mesh) was purchased from CHENYI Refractory Abrasive Co., Ltd. (Gongyi, China). Its components are shown in Ref. [23], where the mass ratio of SiO_2 is 55.06%, and the mass ratio of Al_2O_3 is 44.12%;
2. The waste sawdust with a maximum particle size of 2.5 mm (the mass mean diameter: 0.8 mm) was obtained from Hongyun Timber Factory (Xingtai, China). It was observed that sawdust has micropores and rough structures, as shown in Figure 1 [24];
3. The sodium hydroxide (NaOH , white granulated) was supplied by ZHIYUAN Chemical Reagent Equipment Co., Ltd. (Tianjin, China);
4. The sodium silicate solution (53.07 wt.%, $n(\text{SiO}_2/\text{Na}_2\text{O})$: 2.12) was obtained from TIANLIAN Chemical Co., Ltd. (Langfang, China);
5. The hydrogen peroxide solution (H_2O_2 , 30 wt.%) was used as a foaming agent and produced by YIHENG Chemical Glass Equipment Co., Ltd. (Changsha, China);
6. A soap powder surfactant was used as a stabilizing agent, aiming to reduce the surface tension and increase the stability of the foam to obtain a more homogeneous distribution;
7. The distilled water used in the experiment was obtained by NANDAI Trading Co., Ltd. (Wenzhou, China).

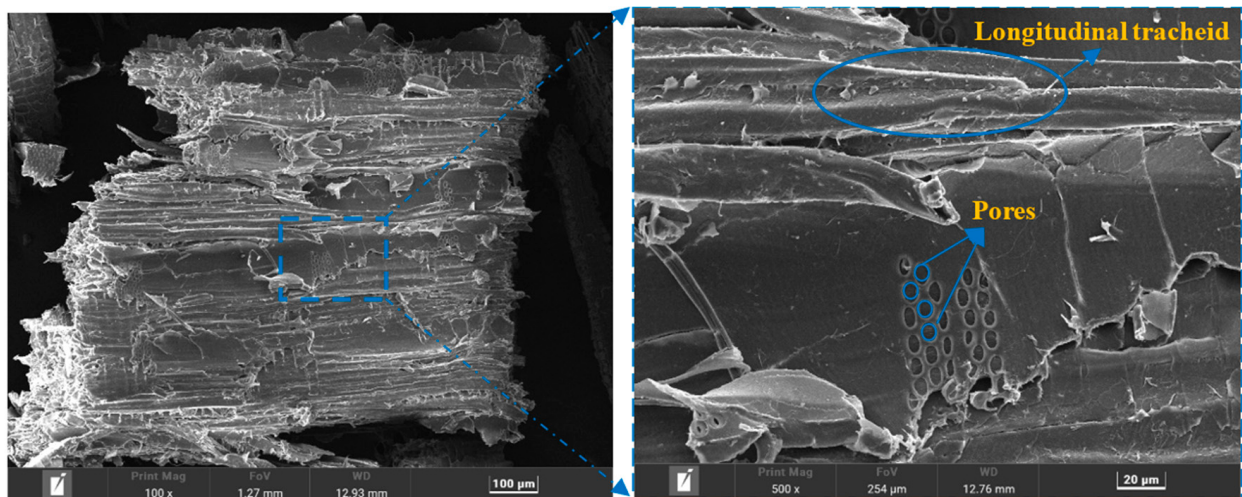


Figure 1. Microstructure and enlarged image of sawdust [24].

2.1.2. Sample Preparation

The preparation process of sawdust/foamed geopolymer adsorbent is presented in Figure 2. The detailed steps were as follows: (1) The NaOH particles and distilled water (mixing water) were added into the sodium silicate in sequence, mixed, and then left at 25 °C for 24 h to stabilize the molecule so as to obtain the alkali activator solution ($n(\text{SiO}_2/\text{Na}_2\text{O}) = 1.32$). (2) The metakaolin was added to the prepared alkali activator and stirred at a speed of 600 rpm for 30 min to obtain the geopolymer slurry. (3) Then, the surfactant and the H_2O_2 were put into the geopolymer slurry in turn and stirred at the speed of 600 rpm for 10 min and 100 rpm for 5 min. (4) A certain amount of distilled water (prewetting water) was added to the sawdust to wet its surface fully, and the prewetted sawdust was mixed with foamed geopolymer slurry at a speed of 200 rpm for 5 min. (5) The mixture slurry was poured into a sealed silicone mold (66 mm * 66 mm * 30 mm), and cured at 25 °C for 24 h and successively at 70 °C for 48 h to consolidate. (6) After the surface of the sample was ground, it was dried at 40 °C for one week to ensure that the sample had limited humidity and moisture content when tested.

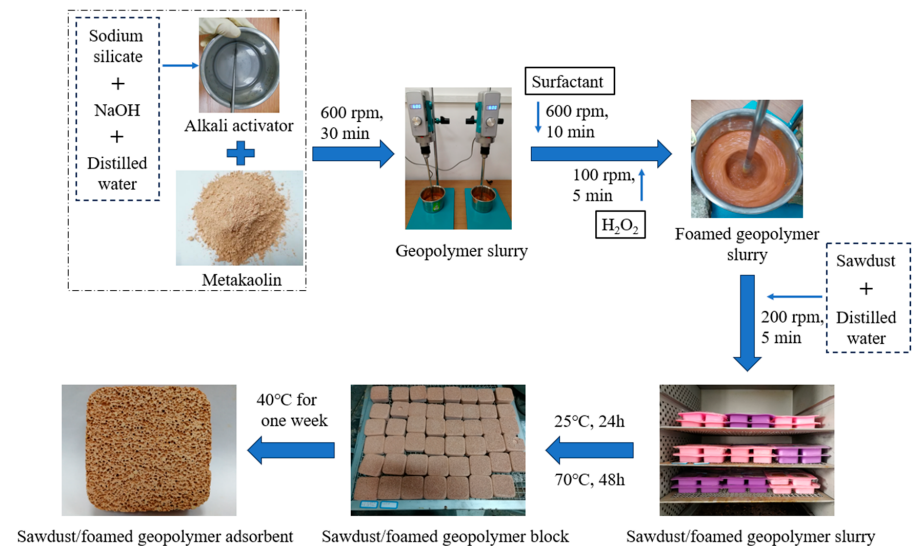


Figure 2. The preparation of sawdust/foamed geopolymer adsorbent.

In this experiment, the detailed mix proportions of sawdust/foamed geopolymer adsorbent are shown in Table 1. The theoretical oxide molar ratios of the geopolymer slurry are: $\text{SiO}_2/\text{Al}_2\text{O}_3 = 3.2$, $\text{SiO}_2/\text{Na}_2\text{O} = 3.3$. The component mass ratios of sawdust/foamed geopolymer adsorbent are as follows: mixing water/geopolymer = 0.5, prewetting water/sawdust = 1.0, surfactant/ H_2O_2 = 1.0, H_2O_2 /geopolymer = 0.014, and sawdust/geopolymer = 0.13. In addition, the above component ratios were obtained from the author's previous studies as the best compromise solution for overall performance [3,24].

Table 1. The detailed mix proportions of sawdust/foamed geopolymer adsorbent.

Sample	Composition (g)							
	Metakaolin	Sawdust	Sodium Silicate Solution	NaOH	Mixing Water	Prewetting Water	H_2O_2	Surfactant
SFG adsorbent	180	38.7	164.7	22.6	58.2	38.7	13.5	4.1

According to the above ratio, the density and compressive strength of the SFG adsorbent prepared were 334 kg/cm^3 and 1.25 MPa, respectively. The total porosity of the SFG adsorbent is 80.4%, of which the open porosity is 75.2%, and the pore size is mainly concentrated in the range of $900 \mu\text{m}$ – $1100 \mu\text{m}$ (Figure 3).

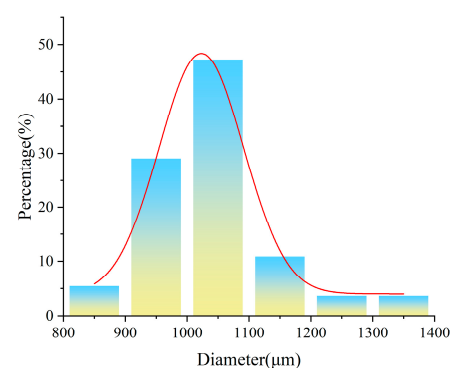


Figure 3. Pore size distribution of sawdust/foamed geopolymer adsorbent.

The microstructure of the SFG adsorbent is given in Figure 4. It can be seen that the interior of porous materials is filled with pores, with thin pore walls and mostly interconnected structures between pores, resulting in a larger specific surface area. In addition, there are cracks on the pore walls of the sample, which are caused by the different thermal properties of sawdust and geopolymers; that is, during the curing process, when the temperature changes, the thermal expansion and contraction of the two materials are not consistent, resulting in the formation of cracks.

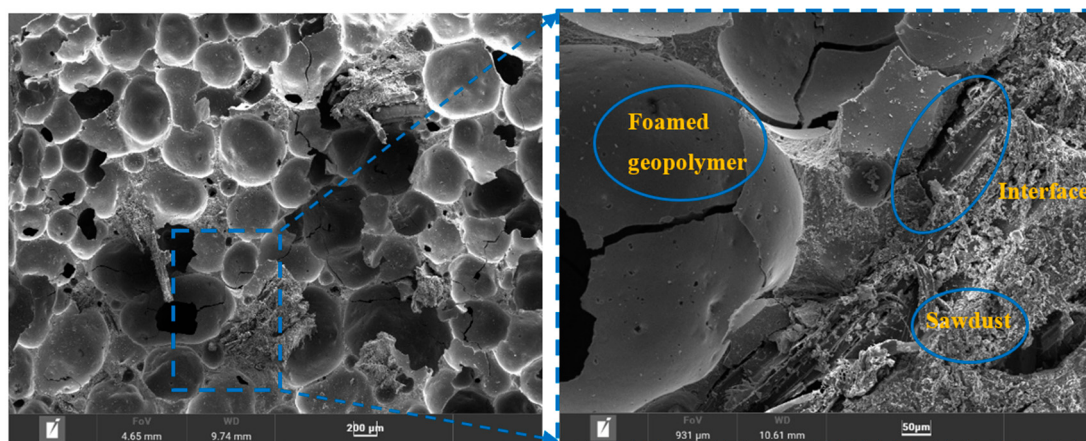


Figure 4. Microstructure and enlarged image of sawdust/foamed geopolymer adsorbent.

2.2. Experimental Methods

2.2.1. Adsorption Tests

The solution containing heavy metal Cu^{2+} ions was prepared by dissolving $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (analytical reagent) into distilled water. Moreover, the pH of the Cu^{2+} solution was adjusted by adding HNO_3 and NaOH solutions. The concentration of Cu^{2+} was tested by using an inductively coupled plasma mass spectrometer (Agilent 7900, Agilent Technologies, Inc., Santa Clara, CA, USA).

The removal rate and adsorption capacity were used as evaluation indicators for adsorption performance. Among them, the removal rate was calculated by the following Formula (1) [25]:

$$\eta = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

where η is the removal rate of Cu^{2+} ions, (%); C_0 is the initial concentration of Cu^{2+} ions, mg/L; and C_e is the residual concentration of Cu^{2+} ions at time, mg/L.

The adsorption capacity was presented in Formula (2) [25]:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (2)$$

where q_e is the adsorption capacity of a unit mass adsorbent for Cu^{2+} ions, mg/g; C_0 is the initial concentration of Cu^{2+} ions, mg/L; C_e is the residual concentration of Cu^{2+} ions at the time, mg/L; V is the volume of the wastewater, L; and m is the mass of adsorbent, g.

In this study, it is necessary to pre-treat the adsorbent before starting the adsorption experiment, mainly to avoid the interference of OH^- leaching from SFG adsorbent prepared by alkali activation technology on metals [21]. The pre-treatment method of the adsorbent is as follows: firstly, the adsorbent material was repeatedly washed with distilled water until the washing water had a neutral pH; then, it was dried at 45°C for 12 h; finally, the adsorbent was left to cool at room temperature. After that, the adsorbents were cut into small blocks according to the requirements (see illustration in Figure 5).

Then, the pre-treated adsorbent was sealed and stored in a container for subsequent adsorption experiments.

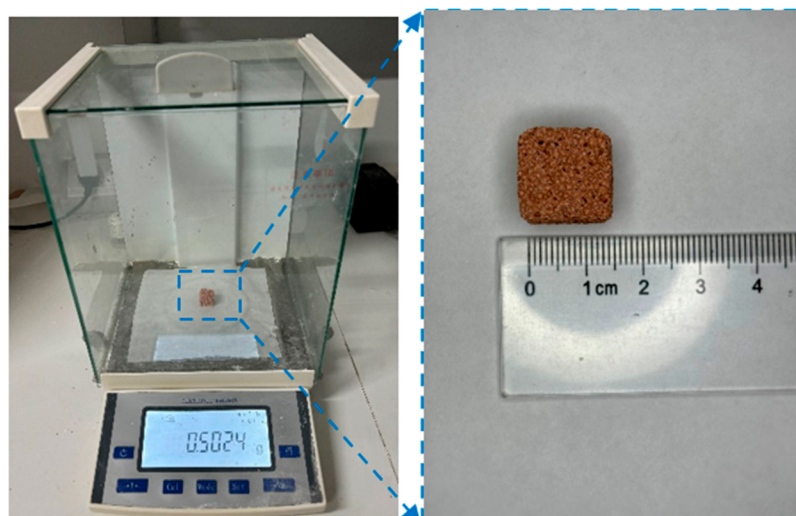


Figure 5. The images of small pieces of SFG adsorbent (taking “ $m_b = 0.5$ g” for example).

The experimental design of the adsorption experiment is shown in Table 2. The effect of five variables on the adsorption process of Cu^{2+} ions by SFG adsorbent is researched, which includes SFG dosage, adsorption temperature, solution pH, contact time, and initial Cu^{2+} solution concentrations.

Table 2. Design of adsorption experiment for sawdust/foamed geopolymers adsorbent.

Sample Number	Temperature (°C)	pH	Initial Concentration (mg/L)	Contact Time (min)	SFG Dosage (g)
D1	25	5	90	720	0.1
D2	25	5	90	720	0.3
D3	25	5	90	720	0.5
D4	25	5	90	720	0.7
D5	25	5	90	720	0.9
T1	25	5	90	720	0.5
T2	30	5	90	720	0.5
T3	35	5	90	720	0.5
T4	40	5	90	720	0.5
T5	45	5	90	720	0.5
P1	25	2	90	720	0.5
P2	25	3	90	720	0.5
P3	25	4	90	720	0.5
P4	25	5	90	720	0.5
P5	25	6	90	720	0.5
W1	25	5	90	60	0.5
W2	25	5	90	120	0.5
W3	25	5	90	240	0.5
W4	25	5	90	480	0.5
W5	25	5	90	720	0.5
W6	25	5	90	960	0.5

Table 2. *Cont.*

Sample Number	Temperature (°C)	pH	Initial Concentration (mg/L)	Contact Time (min)	SFG Dosage (g)
C1	25	5	30	720	0.5
C2	25	5	60	720	0.5
C3	25	5	90	720	0.5
C4	25	5	120	720	0.5
C5	25	5	150	720	0.5

Note: D1–5 denote that the variable is the adsorbent dosage; T1–5 denote that the variable is the solution temperature; P1–5 denote that the variable is the solution pH; W1–6 denote that the variable is the contact time between the SFG adsorbent and heavy metal solution; C1–5 denote that the variable is the initial Cu^{2+} solution concentration.

2.2.2. Characterization Methodologies

The morphology and microstructure of sawdust and SFG adsorbent were characterized using the Stereomicroscope (BD-60 T, Boshida optical instrument Co., Ltd., Shenzhen, China) and field-emission scanning electron microscopy (FESEM, MIRA3 and MIRA4, TESCAN, Brno, Czech).

The density was obtained by dividing the sample mass by its volume. The compressive strength was tested by the compression strength tester (WDW-50, Fangyuan Testing Instruments Co., Ltd., Jinan, China) with a range of 0–50 kN, and the loading rate of 10 mm/min was used for all tests.

The concentration of heavy metal ions was measured by using an inductively coupled plasma mass spectrometer (Agilent 7900, Agilent Technologies, Inc., Santa Clara, CA, USA).

The porosity was obtained by Archimedes' principle via water displacement. The pore size distribution characteristics were determined using image observation statistics, and the Nano Measure software (Version 1.2) was used to process micrographs for quantitative analysis of pore size distribution statistics.

Energy-dispersive X-ray spectroscopy (EDS, Carl Zeiss AG, Oberkochen, Germany) was used to detect the elemental distribution at selected positions, which was observed by a ZEISS Sigma 360 (Carl Zeiss AG, Oberkochen, Germany) scanning electron microscope equipped with energy-dispersive spectroscopy. The elemental weight percentages (Wt%) and their standard deviations (σ) were obtained by statistical analysis of the mapping results of the SEM figures.

The phase compositions of the samples were identified using XRD (Rigaku SmartLab SEBruker, Tokyo, Japan) in the range of 10–80° with a nominal step size of 0.02° and a scanning speed of 5°/min.

3. Results and Discussion

The adsorption performance of SFG adsorbent for Cu^{2+} is not only related to its structure but also influenced by experimental conditions. In this study, the effects of SFG dosage (0.1 g, 0.3 g, 0.5 g, 0.7 g, 0.9 g), solution temperature (25 °C, 30 °C, 35 °C, 40 °C, 45 °C), solution pH (2, 3, 4, 5, 6), contact time (60 min, 120 min, 240 min, 480 min, 720 min, 960 min), and initial Cu^{2+} solution concentrations (30 mg/L, 60 mg/L, 90 mg/L, 120 mg/L, 150 mg/L) on the adsorption efficiency of SFG adsorbents were investigated by experimental methods. In addition, in order to evaluate the adsorption performance of SFG adsorbents prepared for heavy metal Cu^{2+} , the adsorption capacity, removal rate, and compressive strength of SFG were compared with other conventional sawdust and foamed geopolymer adsorbents.

3.1. Effect of SFG Dosage

Figure 6 shows the effect of SFG dosage on the adsorption performance of Cu^{2+} . It can be seen that as the dosage of SFG adsorbent increases from 0.1 g to 0.9 g, the adsorption capacity decreases continuously from 39.06 mg/g to 20.22 mg/g. The reason is that, as shown in Formula (2), although the total Cu^{2+} adsorption capacity increases, the Cu^{2+} adsorption capacity per gram of adsorbent decreases [26]. In other words, the increase in adsorbent dosage is greater than the increase in total Cu^{2+} adsorption capacity; thus, the adsorption capacity decreases.

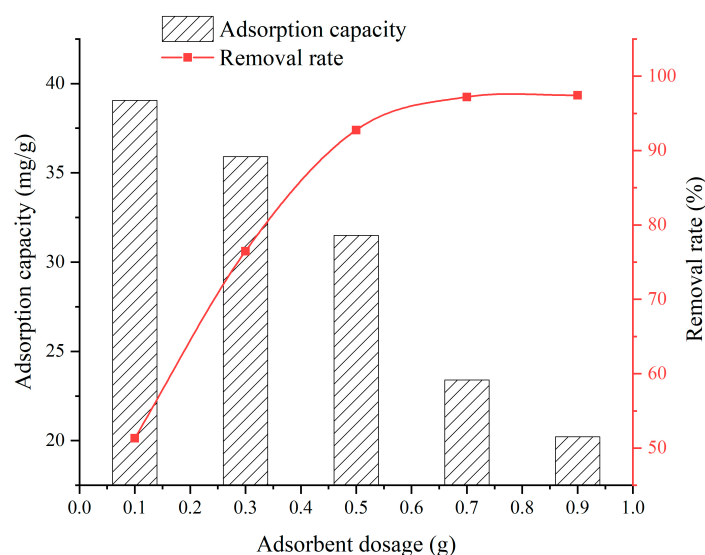


Figure 6. Effect of SFG dosage on Cu^{2+} adsorption.

As shown in Figure 6, with the increase of SFG adsorbent dosage, the removal rate of Cu^{2+} increases first and then gradually stabilizes. This is because the more adsorbent is added, the more adsorption sites it provides, and the corresponding removal rate will continue to increase; however, the Cu^{2+} contained in the solution is fixed, when the amount of SFG adsorbent exceeds the optimal value, the adsorption sites will become vacant, so the removal rate gradually stabilizes.

As a new type of adsorbent, it should not only have high adsorption efficiency but also have the characteristics of green environmental protection and low cost. Therefore, considering the adsorption efficiency and economic benefits, 0.5 g of the SFG was chosen for subsequent experiments.

3.2. Effect of Solution Temperature

Figure 7 shows the effect of different wastewater temperatures (25 °C, 30 °C, 35 °C, 40 °C, 45 °C) on the adsorption performance of Cu^{2+} ions. As the temperature increases from 25 °C to 45 °C, the adsorption capacity and removal rate of Cu^{2+} increase from 31.5 mg/g to 33.6 mg/g and from 92.76% to 97.31%, respectively. It can be noticed that when the temperature increases from 25 °C to 40 °C, the adsorption capacity of the SFG adsorbent and the removal rate of Cu^{2+} increase rapidly; however, when the temperature is above 40 °C, the growth rate of the two parameters becomes slow and tends to stabilize. This may be due to the fact that the rises in temperature intensify the thermal movement of Cu^{2+} in the solution, increasing the possibility of Cu^{2+} entering the pore structure and thus improving its adsorption performance. Additionally, Duan et al. [27] concluded that the adsorption capacity of porous geopolymer adsorbents for heavy metals is enhanced at high temperatures. However, increasing the temperature of the solution will increase the cost of removing heavy metals and cause additional energy consumption. Therefore,

excellent adsorbents should also have relatively excellent adsorption performance at room temperature (25 °C).

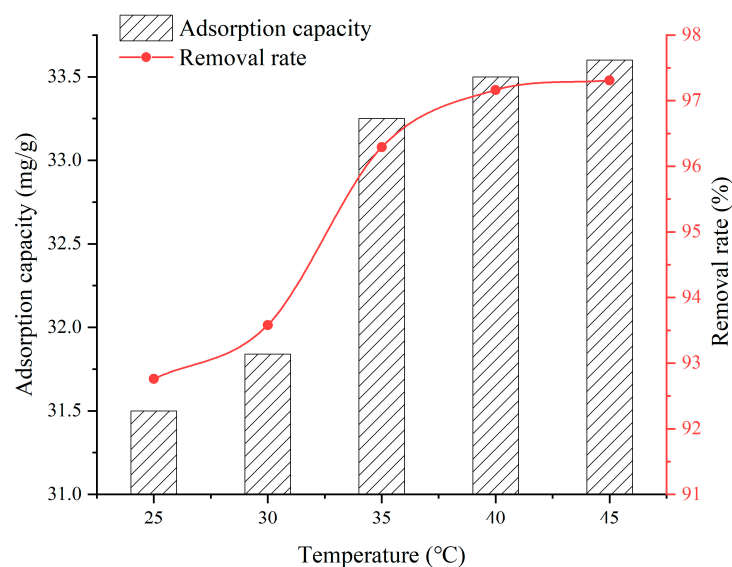


Figure 7. Effect of temperature on Cu^{2+} adsorption.

3.3. Effect of Solution pH

Figure 8 presents the effect of pH values (2, 3, 4, 5, 6) of Cu^{2+} solution on adsorption capacity and removal rate. When the pH value is 2 to 5, the adsorption capacity and removal rate increase from 6.45 mg/g to 31.5 mg/g, and from 58.2% to 92.76%, respectively. This phenomenon can be explained: due to the limited number of active sites on the adsorbent, the higher the pH value of the solution, the lower the H^+ content and the weaker the competition between H^+ and Cu^{2+} , which leads to better access of Cu^{2+} toward the active sites. Therefore, with the increase of pH value, the adsorption performance of the SFG adsorbent is enhanced. However, when the pH value is greater than 5, the adsorption capacity and removal rate begin to decrease. The reason is that the pH of the solution reaches the solubility product of Cu^{2+} , which results in the formation of precipitate on the surface of the adsorbents. The same phenomenon was observed in the work of [28], which indicates that a high pH value will facilitate the formation of $\text{Cu}(\text{OH})_2$ precipitate from Cu^{2+} . Moreover, the precipitate on the surface of the adsorbent blocks the pore structure, preventing further adsorption of Cu^{2+} into the pores; thus, the adsorption performance of the SFG adsorbent decreases.

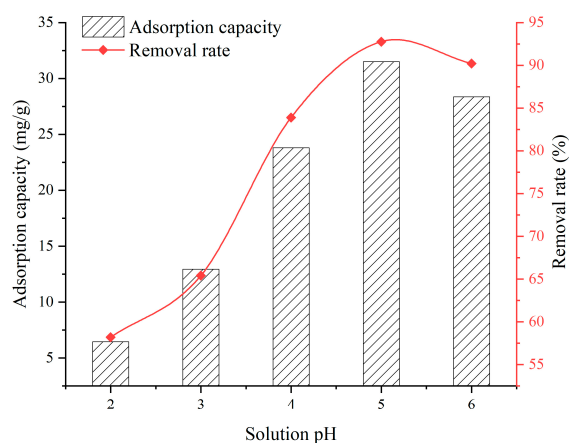


Figure 8. Effect of pH on Cu^{2+} adsorption.

In summary, when the pH value of the solution is 5, the adsorption capacity and removal rate reach their maximum values (31.5 mg/g, 92.76%). Compared with the adsorption performance (24.69 mg/g, 92.8%) of foamed geopolymers for Cu^{2+} studied by Tan et al. [21], the adsorption performance of the novel SFG adsorbent prepared in this study is similar. However, this SFG adsorbent uses sawdust instead of part of the geopolymer, which has the advantages of low price and environmental friendliness.

3.4. Effect of Contact Time

Figure 9 illustrates the influence of contact time (60 min, 120 min, 240 min, 480 min, 720 min, 960 min) on the adsorption process of Cu^{2+} . It can be seen that when the contact time is in the range of 60 min to 240 min, the adsorption capacity and removal rate of Cu^{2+} increased from 9.75 mg/g to 28.6 mg/g and from 38.7% to 87.8%, respectively, with a significant increase. This is mainly because the SFG adsorbent has more active sites in the initial stage of the reaction. And the concentration of Cu^{2+} in the solution is relatively high, while the concentration of Cu^{2+} on the surface of the adsorbent is relatively low, forming a strong mass transfer driving force. Therefore, Cu^{2+} in the solution quickly aggregates on the surface of the SFG adsorbent and occupies the adsorption active sites. When the contact time is greater than 240 min, the growth rate of both is very small. This is mainly due to the decrease in the concentration difference of Cu^{2+} between the solution and the adsorbent surface, which leads to a reduction in mass transfer kinetics, while the adsorption active sites of the adsorbent are occupied, resulting in a slowdown in the adsorption performance of the adsorbent for Cu^{2+} . As time goes by, when the contact time is 720 min, the adsorption capacity and removal rate are 31.5 mg/g and 92.76%, respectively. At this stage, the adsorption process reaches equilibrium, and the adsorption capacity and removal rate no longer increase.

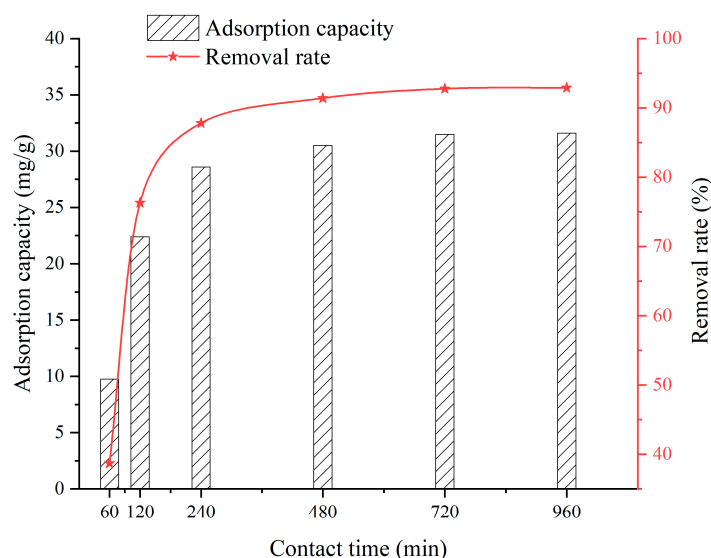


Figure 9. Effect of time on Cu^{2+} adsorption.

3.5. Effect of Initial Cu^{2+} Solution Concentrations

Figure 10 exhibits the effect of the initial Cu^{2+} solution concentration on the adsorption process. As the initial Cu^{2+} concentration increases, the adsorption capacity first rapidly increases and then gradually reaches an equilibrium state. When the initial Cu^{2+} solution concentration increases from 30 mg/L to 120 mg/L, the adsorption capacity increases from 14.52 mg/g to 36.87 mg/g. However, when the initial Cu^{2+} concentration exceeds 120 mg/L, the adsorption capacity no longer increases and remains relatively stable. The reason is that when the initial concentration is low, the required active sites for adsorbing

Cu^{2+} are smaller than those of the SFG adsorbent, resulting in a rapid increase in adsorption capacity; however, as the initial concentration continues to increase, the limited active sites provided by the SFG adsorbent are not sufficient to adsorb all Cu^{2+} [29], so the adsorption sites reach saturation and the adsorption capacity remains constant.

As shown in Figure 10, with the increase of initial Cu^{2+} concentration, the removal rate of Cu^{2+} decreases from 98.96% to 72.3%. When the Cu^{2+} concentration is in the range of 30 mg/L to 90 mg/L, the removal of Cu^{2+} by SFG adsorbent exceeds 90%. However, with the continuous increase of the initial Cu^{2+} solution concentration, the removal rate decreased to 72.3% at a concentration of 150 mg/L. One reason is that at lower concentrations, there is a significant difference in the number of adsorption sites and Cu^{2+} , which allows Cu^{2+} to occupy the activation sites on the SFG surface without restriction, resulting in a higher removal rate; however, as the initial Cu^{2+} solution concentration increases, all adsorption sites are occupied, and there is an excess Cu^{2+} in the solution, leading to a gradual decrease in removal rate. Another reason is that when the initial Cu^{2+} solution concentration is high, physical adsorption becomes the dominant mechanism. Although there is a slight increase in adsorption capacity, the weak bond energy of physical adsorption forces makes the weakly bound ions prone to desorb from the surface and re-enter the solution, leading to a decline in the removal rate.

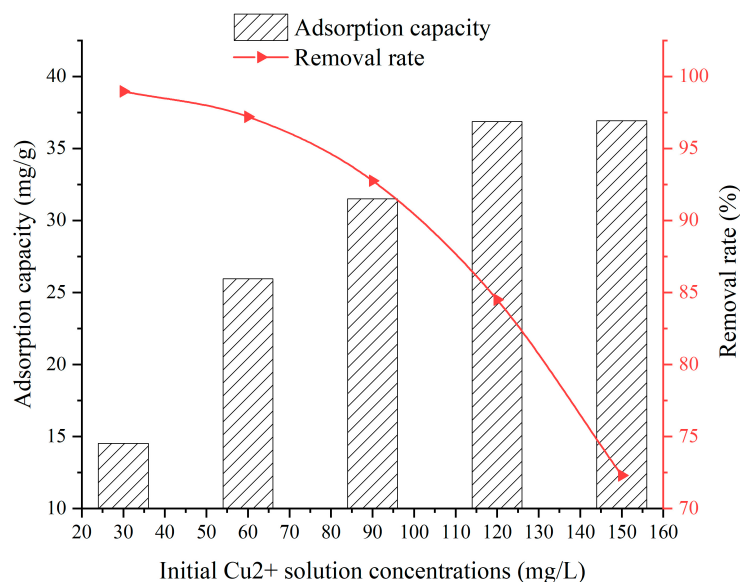
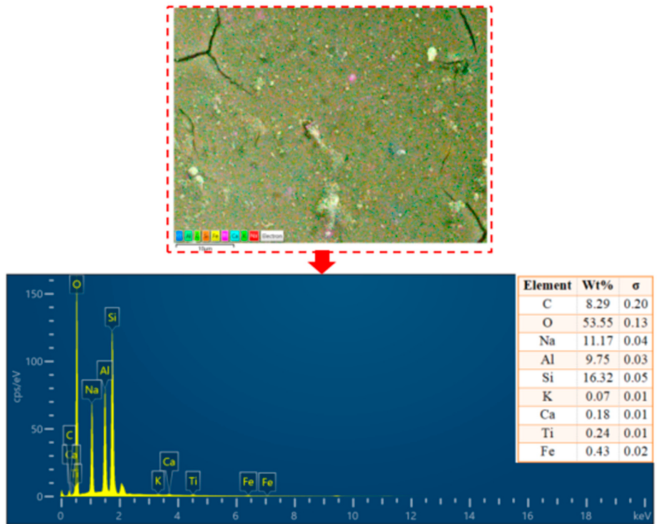


Figure 10. Effect of initial concentration on Cu^{2+} adsorption.

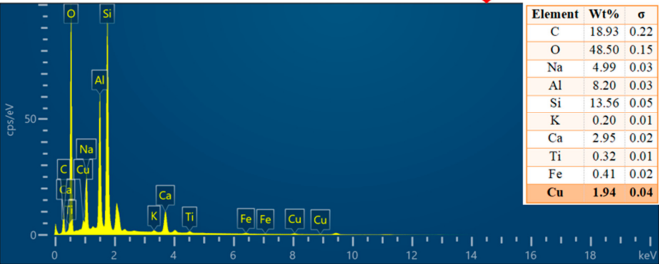
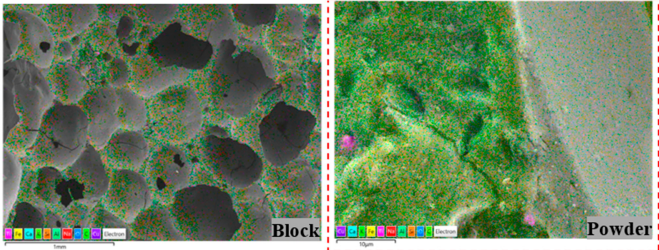
Based on the above experimental results and comprehensive consideration of the adsorption capacity and removal rate of Cu^{2+} , when the initial Cu^{2+} solution concentration is 90 mg/L, it has a good adsorption capacity (31.5 mg/g) and removal rate (92.76%), which means that SFG is a promising adsorbent for removing Cu^{2+} .

3.6. EDS and XRD Analysis

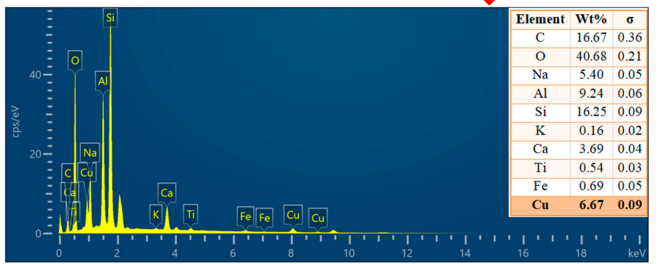
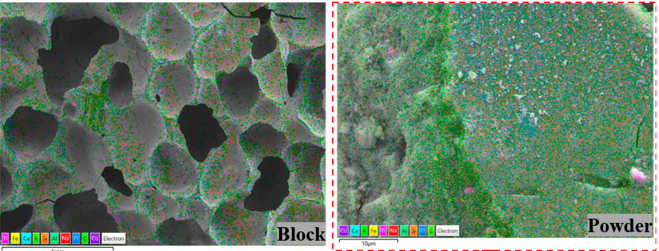
Figure 11 presents SEM images with the EDS elemental analysis of samples at different initial Cu^{2+} concentrations under conditions of 25 °C, pH = 5, SFG dose of 0.5 g, and contact time of 720 min. The basic constituent elements identified by EDS were C, O, Na, Al, Si, K, Ca, Mg, Ti, Fe, and Cu.



(a)



(b)



(c)

Figure 11. Cont.

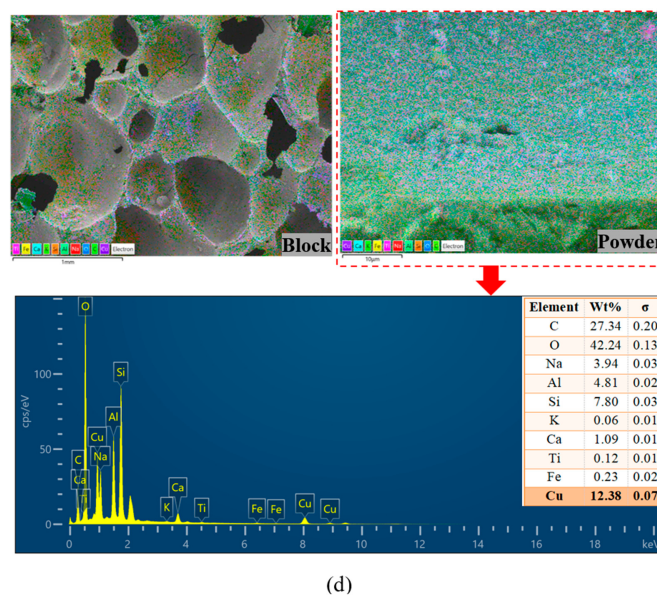


Figure 11. SEM images of (a) original SFG, (b) SFG after adsorption of heavy metals in a solution with an initial Cu^{2+} concentration of 30 mg/L, (c) SFG after adsorption of heavy metals in a solution with an initial Cu^{2+} concentration of 60 mg/L, and (d) SFG after adsorption of heavy metals in a solution with an initial Cu^{2+} concentration of 150 mg/L combined with the elemental analysis from EDS.

Figure 11a shows the EDS spectra obtained after grinding the original block SFG adsorbent into powder. It can be seen that the basic elements contain C, mainly due to the presence of biomass sawdust in the adsorbent; However, no heavy metal Cu was detected in the basic elements, indicating that the raw materials used to prepare the adsorbent do not contain Cu. However, after adsorption, heavy metal Cu was identified in EDS spectra (Figure 11b–d), which proved that the SFG adsorbent can effectively adsorb heavy metal Cu^{2+} in the solution. And the peak of Cu in this study was similar to that reported for the adsorption of Cu^{2+} onto geopolymers [19]. In addition, from the micro-morphology of the block-shaped adsorbent after adsorption in Figure 11b–d, it can be seen that the SFG adsorbent keeps its original shape.

As shown in Figure 11b–d, the block-shaped SFG adsorbent has a large specific surface area and abundant pores, which offer numerous adsorption sites for the heavy metal ions. Meanwhile, ion exchange might have also occurred between the heavy metal Cu^{2+} in the solution and the positive alkaline metal ions (e.g., Na^+ , K^+ , Ca^{2+}) in the geopolymer matrix structure. When the initial Cu^{2+} solution concentration is low (30 mg/L (Figure 11b) and 60 mg/L (Figure 11c)), Cu element is uniformly distributed on the surface of the adsorbent without obvious crystal deposition. Meanwhile, with the increase of initial Cu^{2+} concentration, EDS surface scanning showed that Cu element formed irregular flocs on the adsorbent surface (150 mg/L, Figure 11d). This phenomenon indicates that ion exchange, surface complexation, electrostatic adsorption, and surface adsorption may be the main adsorption mechanisms in the adsorption process of heavy metal Cu^{2+} by SFG adsorbents.

In addition, as the initial Cu^{2+} solution concentration increases from 30 mg/L to 150 mg/L, the Cu element content increases from 1.94 Wt% to 12.38 Wt%. It is preliminarily concluded that the increase of Cu^{2+} concentration has a potential positive effect on the Cu^{2+} adsorption capacity of SFG, and this result is consistent with the conclusion of the experimental test in Section 3.5, which confirmed the adsorption of Cu^{2+} onto SFG.

Figure 12 illustrates the XRD patterns of metakaolin, SFG before and after adsorption. For MK, there is a broad hump between 15° and 30° , indicating the glassy structure of MK. The XRD peak position of SFG is consistent with that of the precursor MK. Minerals such as

anatase [30,31], quartz, kaolinite, and halloysite from the precursors were observed in small amounts in the SFG samples. Compared to MK, the broad weak peak of SFG in the range of 20–30° indicates the presence of amorphous components. The amorphous products are generally N-A-S-H and C-(A)-S-H gels [30]. Furthermore, this also indicates the successful synthesis of geopolymer from MK after the alkali activation process.

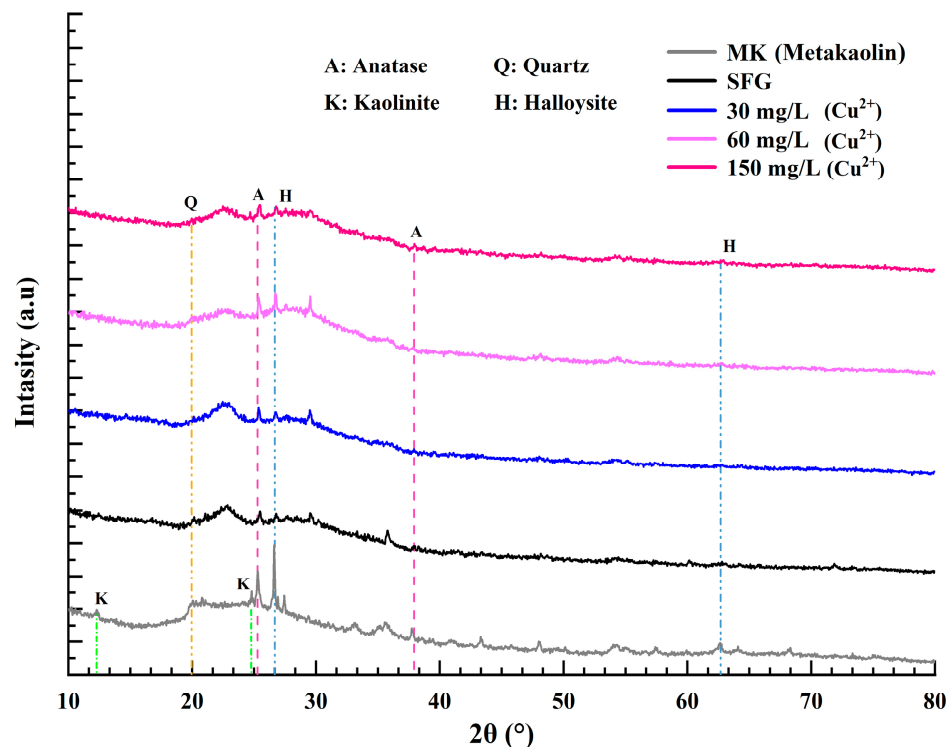


Figure 12. XRD patterns of metakaolin, SFG before and after adsorption.

After adsorption, the primary crystalline phases of SFG remain unchanged. However, the intensity of XRD diffraction peaks slightly weakened, indicating that they may have been affected by the process. Furthermore, it can be seen from Figure 12 that the related XRD pattern search did not reveal the formation of copper precipitates, suggesting that the main mechanism of the adsorption of Cu^{2+} by SFG may not be surface precipitation. However, according to the author's analysis, the following reasons may also affect the observation of diffraction peaks of copper containing substances: (1) The author compared the standard cards of $\text{Cu}(\text{OH})_2$ (PDF#80-0656) and $\text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$ (PDF#42-0746), but their main crystal planes are in the range of 10–30° and are affected by amorphous peaks, resulting in unclear signal observation. (2) SFG adsorbent contains sawdust, which is mainly composed of cellulose, hemicellulose, and lignin; they belong to polymer amorphous materials, which can suppress the exposure of peak signals on the crystal surface of crystalline substances, making it more difficult to visualize peaks. (3) During the adsorption process, precipitates such as copper hydroxide may form; however, the main adsorption mechanism is not chemical precipitation (because the pH value of the experimental test solution is 5), so the precipitation amount is too small, resulting in unclear signal observation.

In summary, through EDS testing, no Cu element was found in the SFG sample, but Cu element was detected in the SFG after adsorption, which also confirms that the SFG sample adsorbs heavy metal Cu^{2+} . Although no Cu-containing crystal structure was found in the XRD test, the gel phase produced by geopolymer reaction was observed in the SFG sample prepared by alkali excitation of metakaolin. In addition, some characteristic peaks also showed slight changes, which further indicates that SFG is involved in the adsorption

process. Therefore, this new SFG adsorbent has certain potential in the field of adsorbing heavy metal Cu^{2+} .

4. Comparison of Adsorption Properties with Sawdust and Foamed Geopolymer

To better evaluate the adsorption performance of novel SFG adsorbent materials for heavy metal Cu^{2+} . The adsorption capacity and removal rate of SFG, conventional sawdust, and foamed geopolymer adsorbents were compared and analyzed, as shown in Table 3. It can be seen that compared to sawdust, the SFG adsorbent has better adsorption performance. According to the work of [32], compared to chemically modified sawdust, the adsorption capacity and removal rate of natural sawdust are lower under the same adsorption conditions. This phenomenon was explained in that the adsorption mechanism of natural sawdust on heavy metal ions is mainly physical adsorption, and the adsorption force is primarily the van der Waals force; while the modified sawdust by alkaline solution has a loose construction and a higher specific surface area, alkali modification increases the -OH groups (this means that the adsorption mechanism involves ion exchange or hydrogen binding) at the same time, thus the adsorption performance of heavy metal ions is significantly improved under the dual action of physical adsorption and chemical adsorption. Additionally, when sawdust is used as an adsorbent, it is difficult to recover due to its powder properties and is prone to secondary pollution.

The SFG adsorbent prepared in this study is comparable to conventional foamed geopolymer in terms of adsorption capacity and removal rate. It can be seen from Table 2 that compared with the foamed geopolymer studied by [21,33,34], SFG adsorbent has a relatively higher adsorption capacity and better compressive strength. Compared with the results of [30], the SFG adsorbent has a better removal rate and compressive strength. Therefore, from an overall perspective, this study utilizes sawdust for high-value resource utilization, which not only solves the environmental problems caused by the combustion of forestry waste but also prepares a new type of high-efficiency adsorbent.

In addition, the existing adsorbents on the market can be divided into inorganic adsorbents, biomass adsorbents, and synthetic polymer resin adsorbents. However, inorganic adsorbents have a small adsorption capacity, are difficult to recover, and can cause secondary pollution, such as zeolites, diatomaceous earth, and kaolin. While biomass adsorbents have poor mechanical strength and are prone to dissolution and fragmentation, synthetic polymer resin adsorbents are expensive, difficult to degrade, and prone to secondary pollution. Therefore, in comparison, the SFG adsorbent prepared in this study has good adsorption performance, lower costs, as well as environmental friendliness and recyclability, which gives it a competitive advantage in the field of green adsorbent materials in the future.

Table 3. Comparison of compressive strength, adsorption capacity, and removal rate of this study with sawdust and foamed geopolymer.

Category	Adsorbent	Type	Compressive Strength (MPa)	Metal Ions	Adsorption Capacity (mg/g)	Removal Rate (%)	Test Conditions	Ref.
Adsorbents from sawdust and foamed geopolymer	Sawdust/foamed geopolymer (SFG)	Block	1.25	Cu^{2+}	31.5	92.76	C_0 : 90 mg/L; pH: 5; T_e : 25 °C; t_c : 720 min; m_b : 0.5 g	This work

Table 3. Cont.

Category	Adsorbent	Type	Compressive Strength (MPa)	Metal Ions	Adsorption Capacity (mg/g)	Removal Rate (%)	Test Conditions	Ref.
Adsorbents from sawdust	Modified sawdust by KOH	Powder	/	Cu ²⁺	7.64	85	C ₀ : 30 mg/L; pH: 4.	[32]
	Natural sawdust	Powder	/	Cu ²⁺	3.88	74		
	Sawdust by calcination	Powder	/	Cu ²⁺	15	/	C ₀ : 50 mg/L; pH: 5; T _e : 25 °C; t _c : 4320 min.	[35]
	Sawdust–chitosan nanocomposite beads	Powder	/	Cu ²⁺	1.8	90.32	C ₀ : 50 mg/L; pH: 5; T _e : 30 °C; t _c : 70 min.	[36]
	Sawdust-based biochar	Powder	/	Cu ²⁺	/	90.8	C ₀ : 50 mg/L; t _c : 720 min.	[37]
Adsorbents from foamed geopolymer	Geopolymer foams	Block	/	Cu ²⁺	5.53~5.93	94.9	C ₀ : 50 mg/L; pH: 5; t _c : 1440 min.	[33]
	Geopolymer foams	Block	0.86	Cu ²⁺	64.9	58	C ₀ : 200 mg/L; pH: 5; T _e : 25 °C; t _c : 1440 min.	[30]
	Foamed geopolymer	Sphere	/	Cu ²⁺	24.69	92.8	C ₀ : 200 mg/L; pH: 5; t _c : 2880 min.	[21]
	Porous geopolymer	Block	0.35	Cu ²⁺	19.59	97.96	C ₀ : 100 mg/L; pH: 5; T _e : 20 °C; t _c : 1440 min.	[34]

Note: C₀: initial concentration; T_e: test temperature (°C); t_c: contact time (min); m_b: the mass (g).

5. Conclusions

In this study, a novel sawdust/foamed geopolymer (SFG) adsorbent with high compressive strength was prepared from sawdust and metakaolin. To analyze the influence of SFG dosage, solution temperature, solution pH, contact time, and initial Cu²⁺ solution concentrations, the adsorption capacity and removal rate of SFG adsorbents were tested. The main conclusions are as follows:

- (1) The novel SFG porous material exhibits excellent adsorption performance in removing heavy metal Cu²⁺. Meanwhile, it has the advantages of high mechanical strength, high recyclability, and high moldability, which allows it to be made into different block or spherical shapes according to user needs;
- (2) When the SFG dosage varies from 0.1 g to 0.9 g, the adsorption capacity and removal rate are 20.22~39.06 mg/g and 51.32~98.09%, respectively. When the solution temperature varies from 25 °C to 45 °C, the adsorption capacity and removal rate are 31.5~33.6 mg/g and 92.76~97.31%, respectively. When the solution pH varies from 2 to 6, the adsorption capacity and removal rate are 6.45~28.36 mg/g and 58.2~90.2%, respectively. When the contact time varies from 60 min to 960 min, the adsorption capacity and removal rate are 9.75~31.6 mg/g and 38.7~92.9%, respectively. When the initial Cu²⁺ solution concentration varies from 30 mg/L to 150 mg/L, the adsorption capacity and removal rate are 14.52~36.92 mg/g and 72.3~98.96%, respectively;

- (3) Considering the economy, environmental friendliness, and comprehensive performance, a desirable SFG adsorbent with an SFG dosage of 0.5 g, temperature of 25 °C, pH of 5, contact time of 720 min, and initial Cu²⁺ solution concentrations of 90 mg/L, is recommended, of which the adsorption capacity is 31.5 mg/g with the removal rate being 92.76%;
- (4) It is feasible to replace a portion of metakaolin with natural sawdust to prepare SFG adsorbent. Although the adsorption performance of the SFG adsorbent prepared is similar to that of foamed geopolymer, this method fully utilizes waste sawdust and has the advantages of low carbon, energy saving, and environmental protection.

The new SFG adsorbent proposed in this paper can effectively adsorb heavy metal Cu²⁺, which indicates that it has a good application perspective in the field of the treatment of heavy metal ions in wastewater. In addition, sawdust from forestry waste was used instead of some metakaolin in this experiment. Therefore, compared with other commercial adsorbents, such as inorganic adsorbents and synthetic polymer resin adsorbents, the production cost of this adsorbent is lower. However, the adsorption capacity still needs to be further improved. The next investigations will focus on the adsorption mechanism of SFG adsorbents and search for better optimization methods by deducing from the mechanism.

Author Contributions: Methodology, H.L. and X.Z.; Investigation, S.W.; Resources, S.W.; Writing—original draft, S.W.; Writing—review & editing, X.Z.; Supervision, H.L. All authors have read and agreed to the published version of the manuscript.

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