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# Removal of copper (II) from mining waste water by adsorption onto activated carbons produced from hazelnut shell

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Abstract: Mining wastewater is one of the most important environmental problems today because of the heavy metal ions they contain. Wastewaters containing heavy metal pollution are generally acidic waters with low BOI (Biochemical oxygen demand) value. Various processes such as mineral processing waste disposal, illegal mining, domestic waste disposal, and others result in the release of heavy metals into the waters. One of the heavy metals that cause problems in terms of environmental pollution is copper. Copper metal is commonly found in metal and metal plating industry wastewater. Although metal industry wastewater is low in quantity, they are toxic waste. For this reason, industrial wastewater must be treated before being discharged into the receiving environment. In this study, the amount of Cu in wastewater of the mining operation in Gümüşhane was determined. Heavy metal pollution was eliminated with an adsorbent produced from agricultural biowaste. It has been tried to determine the amount of adsorbent, contact time, and treatment efficiency of wastewater at different pHs of activated carbon produced by chemical activation. As a result, an average of 88.7% efficiency was obtained with activated carbon activated with KOH, while 22.4% copper removal efficiencies were obtained with raw material (hazelnut shell) used without activation. It was determined that the appropriate working pH was 5.0; the amount of adsorbent was 0.05 g and Qmax 121.2. As a result, in this study, very low-cost adsorbents were produced using waste hazelnut shells, and a biotechnological approach was proposed to clean copper-containing wastewater.

Keywords: Activated carbon, adsorption, chemical activation, wastewater, hazelnut shell

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#### **1** Introduction

Contamination by heavy metals can be in dissolved or suspended forms. The presence of contaminants exceeding permissible limits poses a significant risk of carcinogens in mammals. Copper (Cu) is a metal heavily used in industries such as plating, mining and smelting, brass production, electroplating industries, oil refining and excessive use of Cu-based agricultural chemicals mining (Bozbeyoglu and Doğan 2023). Industries generate wastewater with varying levels of Cu(II) ions, which negatively impact aquatic environments. According to WHO, the exposure limit value of Cu (II) metal in wastewater is 2.0 mg/l. If it exceeds this value, diseases such as kidney failure, allergies and anemia occur in people (Güler et al. 2001; Budak et al. 2022).

The most commonly used techniques for removing Cu(II) ions from wastewater include oxidation, reduction, precipitation, membrane filtration, biological process, ion exchange, and adsorption. Activated carbons are used in various fields including environmental protection, production of fine chemicals and medicines, food processing, and military activities. Among various purification technologies, activated carbon adsorption is widely used due to its porous surface structure, high surface area, non-harmful nature, and ease of use. However, the high cost of activated carbon has led to the development of new adsorbents with similar properties but lower cost.

The aim of the study was to investigate the performance of Cu(II) adsorption by producing activated carbon from hazelnut shells (HS-AC), which are a type of agricultural biowaste. The use of renewable resources such as biomass is crucial for the sustainable development of the social economy. The study aimed to determine critical factors influencing high surface area activated carbon production from hazelnut shells.

#### 2 Materials and Method

#### **2.1 Materials**

#### Preparation of hazelnut shell and mining waste water

Hazelnuts were collected in Trabzon's farm. Raw hazelnut shells were dried in an oven at  $110^{\circ}$ C for 12 h to remove the moisture content. The dried shells were smashed up to a size range of 1–2 mm (with a coffee grinder) pulverized physically and sifted through a 60 mesh sieve. A sample of wastewater was collected from the drainage pool in the mining facility. It was transferred to sterile glass bottles and stored at room temperature away from sunlight until analysis.

#### **Preparation of activated carbon**

The activated carbon was prepared by following steps: (1) 100 g of the selected fraction of hazelnut shell was impregnated with concentrated KOH. (2) Heated the tube to  $750^{\circ}$ C at a rate of  $30^{\circ}$ C/min and kept this condition for 12 h to carbonize the raw material in a hot air oven and then it was washed with distilled water until the pH of the activated carbon reached six, (Fig. 1).(3)The carbonized material was washed with distilled water to remove the free acid. (4) Dried at  $105^{\circ}$ C, then removed after the furnace cooled to room temperature. The carbonized materials were treated with an activator for 30 minutes (Bansal et al. 2005).



#### Fig. 1 HS-AC production process

#### 2.2 Characterization techniques

#### 2.2.1 Ash content determination

Ash content of original hazelnut shells (HS) and hazelnut shells activated carbon with KOH (HS-AC) adsorbents was determined using the ASTM (American Society for Testing Materials) standard method. The crucibles were placed inside the muffle furnace and the furnace was set to gradually increase the temperature to 500°C over the course of 1 hour, and then to 750°C over the course of 2 hours. Once the desired temperature was reached, the samples were removed from the furnace and allowed to cool inside a desiccator. Their weight was then measured (El-Hendawy et al. 2001)

#### 2.2.2 Determination of volatile matter amount

The ASTM standard method was employed to determine the volatile matter contents of adsorbents. To prepare for the

experiment, the platinum crucible was first placed in a muffle furnace along with its lid and heated to 950°C for 30 minutes. After that, 1 gram samples were added to the crucibles, and the crucibles with their contents were kept in the muffle furnace at 950°C for a further 10 minutes. The samples were removed from the oven, cooled in a desiccator and weighed.

#### 2.2.3 Boehm Titration

With Boehm titration, surface functional groups of various adsorbents and especially activated carbons can be detected (Boehm 1966). To quantify the number of lactonic, phenolic, and carboxylic groups in mmol for both adsorbents through Boehm titration, 0.05g of adsorbent was treated with 50mL of 0.1N NaOH, NaHCO<sub>3</sub>, and Na<sub>2</sub>CO<sub>3</sub> separately. The mixture was then shaken at 400rpm for 24 hours.Total surface acidic groups (lactonic, phenolic, and carboxylic) were all neutralized with 0.1 N NaOH and determined. After shaking and filtration, the filtrate was titrated with 0.1 N HCl (Duman et al. 2009).

#### 2.2.4 pH and pH at neutral load point (pHpzc)

0.1 g of adsorbent was mixed with a 0.1 M NaCl solution. The mixture was shaken for 24 hours at pH values ranging from 2 to 10 to determine pHpzc values of the adsorbent. As a result of shaking, the adsorbent and the solution were separated from each other by centrifugation, and the equilibrium pH values of the solutions were measured with a pH meter. The point where the graph cuts the x-axis from the y-zero value is recorded as pHpzc (Duran et al. 2011).

#### 2.2.5 FT-IR analysis

Thoroughly dried adsorbent samples (few mg) were measured before and after adsorption using direct spectra in the FT-IR device within the 200-4000 cm<sup>-1</sup> wavelength range.

#### 2.3 Equilibrium, kinetic and thermodynamic parameter

For the characterization of HS and HS-AC the effects of pH, contact time, temperature, and amount of adsorbateadsorbent as adsorption tests from aqueous solution were investigated in conjunction with equilibrium, kinetic, and thermodynamic parameters (Bozbeyoglu et al. 2020).

#### 2.4 Adsorption tests

Optimum pH values were determined on activated carbons. pH values of metal-containing mining wastewater were adjusted to pH 5.0 for Cu. Then, 10 mL of each of these solutions (5.0 g/L activated carbon suspension), whose pH was adjusted, were treated separately with 0.05 g of activated carbons for 6 hours. After the completion of the process, the metal concentrations in the solutions filtered with a 0.45  $\mu$ m nitrocellulose membrane through a vacuum filtration device were determined using MP-AES. Each trial was performed in at least three parallels, and the averages of the results were given. 1g with the help of the remaining adsorbate concentration (*Ce*) without being adsorbed in the solution (Gündoğdu 2010; Bozbeyoglu 2020).

The amount of adsorbate adsorbed by 1 g of adsorbent is calculated in mg/g with the following formula:

$$Qe = \frac{(Co - Ce) \cdot V}{m}$$

Percent adsorption was also calculated with the following formula:

Adsorption(%) = 
$$\frac{Co - Ce}{Co} \cdot 100$$

Qe: The amount of adsorbate adsorbed by 1 g of adsorbent (mg/g)

*Co* : Initial adsorbate concentration (mg/L)

Ce : The amount of adsorbate remaining without being adsorbed in solution at equilibrium (mg/L)

V: Adsorbate volume (mL)

m: Amount of adsorbent (g)

MA : Atomic or molar mass (g/mol)

#### **3 Results**

#### 3.1 Characterization Analysis Results

## Moisture, volatile matter, fixed carbon and ash content results

The results obtained regarding moisture, ash, volatile matter, fixed carbon analyses, and production yields of this HS-AC and original HS are given in Table 1. High moisture adsorption of activated carbons is normally undesirable. The activated carbon produced has a higher moisture content compared to the starting material HS. It is expected that  $H_2O$  molecules are trapped inside the pores of HS-AC, due to its more porous structure compared to HS. When examining the volatile matter content of adsorbents, HS, the starting material, has the highest amount of volatile matter, as expected. Since no heat treatment is used, HS contains high levels of volatile organic compounds.

Table 1. Brief analysis results of adsorbents

Parameters (%)									
Sample	Moisture	Volatile matter	Fixed carbon	Ash	yield				
HS	4.7	79.2	11.9	4.2	-				
HS-AC	15.1	21.7	53.0	10.2	75.3				

#### **Elemental Analysis Results**

Table 2 shows the results of the elemental analyses performed on the adsorbents using the Leco CHNS 932 elemental analyzer. The oxygen amounts presented in the table are calculated based on the difference. The analysis revealed that the fixed carbon amounts of the produced activated carbon are higher than those of the HS-AC (Akyıldız 2007). During the production of activated carbon, hydrogen, and oxygen are separated from the structure along with volatile components in the gas phase.

 Table 2. Element Contents of Adsorbents (%)

Element Contents (%)								
Sample	С	Н	Ν	S	*0	**C/H		
HS	47.6	6.23	0.87	0.14	49.70	7.64		
HS-AC	58.53	2.96	0.28	-	38.23	19.77		

<sup>\*</sup>Calculated from difference.\*\*Mole ratios-Not determined

#### Determination of pH and pHpzc

Figure 2. shows the graph of equilibrium pH values versus initial pH of adsorbents. It is observed that the initial pH values slightly change after 24 hours of treatment in the unbuffered system. In other words, since the buffer system is not used when adjusting the initial pH, the surface functional groups of the adsorbents can easily interact with the  $H^+$  and  $OH^-$  ions in the solution.



**Fig. 2** Initial pH vs. equilibrium pH plot for pHpzc determination (variation between initial pH and equilibrium pH)

#### Scanning electron microscopy (SEM) analysis

The surface of the adsorbent was characterized by scanning electron microscopy (SEM, Philips XL30S-FEG). It interacted with the hazelnut shell in chemical activation, as seen from the porosity after treatment with potassium hydroxide (Condon 2006). Although the porosity is homogeneous, deep enough pores are not formed, resulting in low surface area. HS-AC has a significant number of pores that trap and adsorb metal ions (Fig. 3).

The fact that the pore walls are thicker than other activated carbons can be explained by the reaction of potassium with the organic structure. Pore width (nm) for HS and HS-AC was measured by BET (Brunauer–Emmett–Teller nitrogen adsorption technique) (Fig. 4)





Fig. 3 SEM images for (a) HS and for (b) HS-AC



Fig. 4 Pore width (nm) for (a) HS-AC and for (b) HS

#### **Boehm titration**

The results from the Boehm titrations for determining surface functional groups are presented in Table 3. As can be seen from the table, the produced activated carbon has rich acidic groups on its surface.

Table 3. Quantitative amounts of surface	acidic groups by Boehm
Titration for adsorbents	

	Acidic groups (mmol/g)						
Adsorbent	Total acidic groups	Carboxylic groups	Phenolic groups	lactonic groups			
HS	2.63	1.90	0.41	0.32			
HS-AC	6.30	1.74	3.24	1.32			

Iodine and methylene blue numbers

The iodine number measures microporosity, referring to 0-2 nm pores. A relatively high iodine number also indicates that the relevant adsorbent has a high surface area (especially high microporosity) (Döşemen 2009). The methylene blue number is a measure of the ability of the adsorbent to adsorb larger organic molecules. Increasing surface area, porosity, and active groups enhance iodine and MB adsorption (Sun 2010). Figure 5 gives a comparison of iodine numbers and MB numbers of adsorbents.



Fig. 5 Comparison of I2 and MB numbers of adsorbents

FTIR and XRD analysis results



Fig. 6 IR Spectrum for HS-AC

When the IR spectrum for HS-AC is examined in Figure 6; The slightly broad peak appearing at 3691 cm<sup>-1</sup> is due to the moisture and/or hydroxyl (<sup>-</sup>OH) groups contained in the sample. Hydroxyl containing groups; phenols, alcohols and carboxylic structures. It is expected that these groups already exist within the original Central Committee, but they undergo deformation at high temperatures during activated carbon production, except for HS-AC (Fig. 6). The peaks appearing at 2972 and 2307 cm<sup>-1</sup> belong to aliphatic C–H bonds. The peak appearing at 1740 cm<sup>-1</sup> is the carbonyl (C=O) stretching peak.

#### **XRD and BET anaysis results**

As can be seen from Fig. 7, the major peak associated with Na at  $2\theta = 25^{\circ}$  for hazelnut shell ash, also peaks of Ca, K, P, KCl, CaCl<sub>2</sub>, KCl are found according to the library data of the XRD instrument. The hazelnut shell ash primarily consists of silica. XRD pattern shows amorphous silica structure at  $2\theta = 20^{\circ}$ . All the carbon samples have two broaden peaks around  $2\theta = 22^{\circ}$  and  $2\theta = 46^{\circ}$  which indicates (002) and (001) planes of graphitic structure. These peaks are characteristics of amorphous carbon with a turbostratic structure. This means that graphite-like microcrystallites are bounded by a crosslinking network of several graphite-like lavers, with each laver having a random direction. Furthermore, it is worth noting that low intensity and wide peaks indicate that some graphite-like amorphous structures have been formed and the sharp and narrow peaks show that highly ordered graphite-like structures.



Fig. 7 XRD Spectrum for HS-AC

 Table 4. BET surface areas and pore size distributions of adsorbents

	SBET	S <sub>mikro</sub>	,	Sme	20	$V_{toplam}$	V <sub>mikro</sub>	)	V <sub>mezo</sub>		$D_{\mathrm{p}}{}^{\mathrm{a}}$
Ads	m²/g	m²/ g	%	m ²/ g	%	cm <sup>3</sup> /g	cm <sup>3</sup> /g	%	cm³/ g	%	nm
HS	0.12	0.12	100	-	-	0.003	0.03	100	-	-	-
HS- AC	598.6	-	-	-	-	0.32	0.27	83.6	0.05	16.4	2.12
SBET : BET surface area Smicro: Micropore surface area											

SBET : BET surface area Smeso: Mesopore surface area diameter

Vtotal: TotalDp : Average pore - : Could not be determined HS has a mostly microporous structure, while HS-AC is more mesoporous.

### **3.2** Adsorption analysis results and equilibrium, kinetic and thermodynamic parameters

### **3.2.1** Effect of initial Cu(II) concentration and adsorption isotherms

Adsorption of Cu (II) from aqueous solution on the adsorbent fixed amount to investigate the effect of the initial Cu(II) concentration. A serial Cu solution in the range of adsorbent (1.0 g/L) and 50–1000 mg/L under optimum conditions was treated. The Ce-Qe graph drawn in the light of the data shows that a rapid increase in adsorption at the beginning that the increase slows down and progresses towards reaching equilibrium (Fig. 8). In this study, the Langmuir and Freundlich isotherm models were used to analyze the results.



**Fig. 8** Effect of initial Cu(II) concentration and adsorption isotherms: (a) *Ce* vs *Qe* graphs, (b) *Ce* vs *Ce/Qe* graph for Langmuir isotherm (c) Ln(Ce) vs Ln(Qe) for Freundlich isotherm, graph for Cu(II) adsorption on HS-AC (Initial Metal conc: 50-1000 mg/L, pH: 5.0, HS-AC dose: 1.0 g/L)

This theoretical equilibrium isotherm, developed by Langmuir, accepts that there are receptor points on the surface of the adsorbent and that each receptor point can adsorb only one molecule, so the resulting layer will be one molecule thick.

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_{maks}} + \frac{1}{bQ_{maks}}$$

Qe: Amount of adsorbate adsorbed by 1 g of adsorbent (mg/g)

Qmax : Maximum monolayer adsorption capacity (mg/g)

Ce: The amount of adsorbate remaining unadsorbed in the solution at equilibrium (mg/L)

b: Constant related to free energy or adsorption enthalpy (L/mg)

It is often observed that the Freundlich isotherm is followed in situations where there is multilayer adsorption. The equation of the model expressing the change of the amount of substance (Qe) adsorbed by the adsorbent with pressure and concentration is expressed below (Freundlich 1906);

 $Qe = Kf \times Ce 1/n$ 

 
 Table 5. Langmuir and Freundlich isotherm constants obtained for Cu (II) adsorption

Cu (II)	Langmu	uir consta	Freun consta				
Ads	Qmaks (mg/g)	Qmaks (mmol /g)	b (L mg <sup>-1</sup> )	$\mathbb{R}^2$	$\begin{array}{c} K_{\rm f} \\ (mg \\ g^{-1}) \end{array}$	n	R <sup>2</sup>
HS	54.4	1.80	0.0129	0.9879	17.54	3.72	0.9979
HS- AC	121.2	1.91	0.0346	0.9984	30.96	4.81	0.9667

The Langmuir and Freundlich adsorption equations were used to expiries adsorption phenomenon of the Cu (II). The Freundlich ( $R^2$ : 0,9979) isotherm model at those optimum conditions best defined the equilibrium data of HS but for the HS-AC is suitable for the Langmuir isotherm model ( $R^2$ :0,9984)

### **3.2.2** Effect of temperature on adsorption of Cu(II) on activated carbon and adsorption thermodynamics

Qe graphs drawn against the studied temperature values are given in Fig. 6. As can be seen, the adsorption behavior of Cu ions on the relevant adsorbents proceeds through an endothermic mechanism. To put it simply, the adsorption efficiency is enhanced by higher ambient temperatures. Additionally, the thermodynamic constants of adsorption were assessed. The negative value of  $\Delta G^{\circ}$  confirms the spontaneous nature of the adsorption process. The positive value of  $\Delta S^{\circ}$  shows that the randomness at the solid solution interface increases during adsorption, while the positive value of  $\Delta H^{\circ}$  indicates that the adsorption process is endothermic.

Thermodynamic data for Cu(II)								
Adsorbent	Т (°С)	Kd	ΔG° (kj/mol)	ΔS° (J/mol.K)	ΔH° (kj/mol)			
HS	5	0.66	0,96	22.37	7.11			
	15	0.77	0,63					
	25	0.85	0,41					
	35	0.94	0,16					
	50	1.01	-0,03					
HS-AC	5	1.82	-1,38	32.77	7.67			
	15	2.10	-1,78					
	25	2.41	-2,18					
	35	2.59	-2,44					
	50	2.89	-2,85					

Table 6. Thermodynamic data for Cu(II)

The effect of temperature Cu (II) uptake capacity onto HS-AC was studied at 100 mg/L initial metal ion concentration.. Adsorption studies were carried out for 24h and it was observed that, the amount of adsorbed Cu (II) increased linearly with time at the beginning of adsorption. A larger amount of Cu (II) ions was removed in the first 10 min of contact time and the equilibrium was established in 30-60 min at the end of a rapid adsorption for all temperatures studied. After a period of 120 minutes, the adsorption of Cu (II) reached equilibrium and there was no further adsorption. The optimal temperature for Cu (II) adsorption was found to be  $60^{\circ}$ C. Removal of copper (II) ions increased with temperature up to  $60^{\circ}$ C.



**Fig. 9** Effect of Temperature: Temp. vs Qe graph and (Initial Cu conc.: 250 mg/L, pH: 5.0, HS-AC dose: 5.0 g/L)

### **3.2.3 Effect of contact time and adsorption kinetics on the adsorption of Cu(II) ions on activated carbons**

Adsorbents in amounts of 5.0 g/L were treated with solutions with pH values of 5.0 for Cu (II) ions, for periods of 0–12 hours. The solutions collected at specific intervals using a micropipette were determined via MP-AES, and the remaining amounts were calculated. Here, the amount of metal loaded per gram of adsorbent was calculated (mg/g). Qt graphs drawn from the obtained data against time are shown in Fig. 10. From the figure, it is evident that the time required to attain equilibrium for all metal ions is quite short, and the process is completed in almost two hours. However, to ensure complete balance for all metal ions, it was decided to extend the processing time to four hours (Mafra 2013).



**Fig. 10** Effect of contact time and kinetics on adsorption of Cu by HS–AC (Initial pH: 5.0, initial Cu concentration: 250 mg/L, HS-AC dose: 5.0 g/L, agitation time: 12 h)

Various kinetic models namely the lagergren's pseudo-first order, pseudo-second order, and intraparticle diffusion models have been used for the validity of the experimental adsorption process for Cu (II) onto HS-AC. The HS-AC can be used as an effective low-cost agricultural waste adsorbent for the removal of Cu (II) with HS-AC's adsorption capacity QMax: 121.2 mg/g at 60°C. The kinetics of Cu (II) adsorption onto HS-AC was examined using the pseudo-first and pseudo-second order kinetic models. The results indicated that the Intraparticle diffusion equation provided the best correlation of the sorption data. The values of various kinetic parameters are tabulated in Table 7. The results showed that, the correlation coefficient for the Intraparticle diffusion model obtained 20-60°C was very low and they increased with increasing temperature.

The pH of an aqueous solution is an important controlling parameter in the process of adsorption. The adsorption capacity of copper (II) ions by HS-AC was analyzed in a pH range of 2-12. The study found that removal of copper (II) ions increased as solution pH increased, and reached a maximum at an equilibrium pH of about 4 when the initial copper (II) ion concentration was 100 mg/L. Experiments were carried out up to pH value of 5 due to the fact that metal precipitation occurred at higher pH values. At very low pH values (pH=2-3), copper (II) ion adsorption was found to be very low due to competition between H<sub>3</sub>O<sup>+</sup> and copper (II) ions for the adsorption sites. In addition when the pH increases, there is a decrease in positive surface charge, which results in a lower electrostatic repulsion between the positively charged metal ion and the surface of HS-AC, favoring adsorption. The study revealed that the loading of copper (II) ions onto HS-AC increased until pH 4 and then remained constant until pH 5. Beyond pH 6, hydroxide precipitations occurred. These precipitations, along with adsorption, contributed to the removal of metal ions.

 Table 7. Kinetic data for adsorption of Cu(II) ions from wastewater

 on HS-AC

	Constants	HS	HS-AC
Cu(II)	Qe (den), mg/g	23.20	31.60
	k1, dak <sup>-1</sup>	8.17	11.33
Pseudo 1st order kinetics	$R^2$	0.01185	0.01151
	Qe (hes). mg/g	0.8885	0.8505
	Qe (hes), mg/g	23.42	31.93
Pseudo 2nd order kinetics	$R^2$	0.00688	0.00448
	<i>K</i> id, mg/g.dak <sup>1/2</sup>	16.39	20.11
	<i>C</i> , mg/g	0.577	1.042
Intraparticle diffusion	$R^2$	0.9998	0.9998
	$k1.  \mathrm{dak}^{-1}$	0.7655	0.8135

3.2.4 Effect of pH on the adsorption of Cu(II) ions on activated carbons

#### 4 Discussion

By examining the performance of activated carbon produced by potassium hydroxide activation, it has been determined in studies that hazelnut shell wastes thrown into the environment, can be used as an adsorption material. Thus, a biotechnological approach was created by cleaning the wastewater of mining enterprises that pollute the environment with another waste. In our study, the optimum pH range for Cu metals determined to be around 5, the amount of adsorbent decreased, the agitation time increased, the adsorption capacity increased, The results showed that, the correlation coefficient for the Intraparticle diffusion kinetic model obtained 20-60°C was very low and they increased with increasing temperature. It is seen that the Freundlich isotherm model is more suitable for the adsorption of metals on HS, and the Langmuir isotherm model for Cu (II) adsorption on HS-AC. These results showed that activated carbon can be produced from hazelnut shells by chemical activation with KOH, and that this method is a cost-effective and energy-saving method and can help produce activated carbon in a cleaner and greener way.

#### **5** Conclusion

Since hazelnut shells, which are completely agricultural biowaste, were used as material in this study, activated carbon, which was consumed and imported in Turkey, was produced and a local and natural adsorbent was synthesized. This process will significantly decrease the production cost, which is currently the biggest obstacle to the widespread use of activated carbon. Furthermore, it will enable large-scale production of activated carbons using agricultural biowastes and pave the way for their use in various agricultural and daily-life applications.

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