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Adsorption of Cd(II) from wastewater using spent coffee grounds by Taguchi optimization

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ABSTRACT

This study evaluated the feasibility of employing spent coffee grounds (SCG) as adsorbent for removing Cd(II) from wastewater. The Taguchi experimental design was applied to determine the optimum adsorption condition. The controllable factors of the treatment procedure of SCG, initial concentration of Cd(II), SCG dose, contact time, and temperature were optimized. The contribution of each controllable factor was also explored. The results showed that the influencing degree of the controllable factors of Cd(II) removal in decreasing order was: concentration of Cd(II) > dose of SCG > treatment procedure of SCG > temperature > contact time. On the other hand, the Cd(II) removal efficiency of SCG in descending order was degreased by solar energy > washing by boiling DI water > washing by DI water; namely, the SCG degreased by solar energy was more effective to adsorb Cd (II) than the other two. Moreover, both results of Langmuir and Freundlich adsorption isotherms adopted to model the equilibrium adsorption data also showed that solar energy degreased SCG was the most effective adsorbent for Cd(II) with the maximum adsorption capacity of 5.46 mg/g.

Keywords: Taguchi method; Adsorption; Spent coffee grounds; Cadmium; Solar energy

1. Introduction

Wastewater from welding, smelting, electroplating, photography, and alkaline batteries usually contains heavy metals, such as Cd(II) [1,2]. Cd(II) is known to be harmful to both environment and human health. Hence, wastewater containing Cd(II) is prohibited from discharging directly into rivers and oceans in Taiwan unless the effluent meets the regulation strictly enforced by Taiwan's effluent standard of 0.03 mg/L.

Traditional methods for heavy metal removal from wastewater include chemical precipitation, ion exchange, membrane filtration, electrolysis, and adsorption using activated carbon. Recently, removal technology of heavy metal ions by biosorption using agricultural waste is getting popular because of the need for further environmental protection [3]. As examples, Cd(II) adsorption by agricultural waste orange peel powder [4], Cr(VI) adsorption by chemically modified coir pith [5] and fruit peel of Litchi [6], Cu(II) adsorption by biochars from crop straws [7] and gooseberry fruit [8] were investigated. In addition, Cd(II), Zn(II), Pb(II), and Cu(II) adsorptions by coffee beans or spent coffee grounds (SCG) were evaluated [3,9,10].

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In this study, SCG is of particular interest because it is known to be a good adsorbent. Further, it is produced daily by a large amount and poses a great environmental concern for its disposal by landfilling. In general, for adopting coffee as an adsorbent, certain pretreatment processes are applied, such as phosphoric acid activation [11–13], zinc chloride activation [10,13], degreasing by ethanol [3] or by NaOH [14]. However, these pretreatments can also cause environmental concerns. Hence, in this study, the SCG treated by washing in cold/boiling DI water or degreased by solar energy was taken as the adsorbent. In order to optimize the adsorption process, Taguchi method was applied for the adsorption of Cd(II).

The Taguchi method established by Genichi Taguchi has been generally adopted to optimize the design variables because it can significantly reduce the overall testing costs and time. The method uses a specifically designed orthogonal array consisting of controllable parameters and their variation levels to optimize experimental conditions [15]. It has been used in wastewater treatment such as modified mesoporous carbon for heavy metals adsorption [16], guava seed carbon for acid orange 7 [17] and acid orange 8 [18] adsorption, zeolite fixed bed reactor for color removal [19], and vacuum membrane distillation for phenolic removal [20].

In this study, SCG and Taguchi methods were combined to optimize the adsorption of Cd(II) as this approach could save time and cost and were rarely presented in the literature. The goal was to obtain optimum operating conditions to effectively use SCG for industrial applications. Moreover, the applicability of both Langmuir and Freundlich adsorption isotherms was also examined.

2. Materials and methods

2.1. Preparation of SCG as an adsorbent

The SCG was obtained from a local coffee maker who prepared coffee by steam extraction of coffee grounds. The collected SCG was divided into three parts, first, washed for five times by DI water (50 mL water per g coffee grounds), second, washing for five times by boiling DI water (50 mL boiling water per g coffee grounds), and last, degreased by solar energy (about 190–200℃, 5 d), which are designated by DI, boiling DI water rinsing (BDI), and degreased by solar energy (DS), respectively, in Table 1. The treated SCG was further dried in an oven at 105℃ for 24 h to remove moisture.

2.2. Experimental methods

The key ideas behind Taguchi method are the orthogonal array for experimental design and signal-to-noise ratio (S/N) for quality determination. The orthogonal array consists of the controllable factors and experimental combinations of equal probability. The controllable factors considered in this study were the treatment procedure of SCG, initial concentration of Cd(II), SCG dose, contact time, and temperature. They were denoted by P, C, D, t, and T, respectively. Each controllable factor had three testing conditions (represented by levels 1, 2, and 3, respectively, as commonly done in Taguchi method) as illustrated in Table 1. Specifically, they were 2.5, 5, and 10 mg/L for C; 1, 2, and 3 g/L for D; 60, 120, and 180 min for t; 25, 40, and 55℃ for T. The stock solutions of Cd(II) were from Merck (Germany).

For the arrangement of Table 1, a full factorial experimental design of five factors with three levels each would require a total of 3^5 (243) experiments. The number of experiments could be reduced greatly by the orthogonal array. Various special designed arrays such as L9 (nine experiments) and L18 (eighteen experiments) were available. Considering the complexity and resources, the orthogonal array of L18 was chosen in this study as shown in Table 2. Hence, the number of experiments was reduced from 243 to 18, a great reduction in time and cost. The numerals 1, 2, and 3 in Table 2 represented levels 1, 2, and 3, respectively.

Table 1
Controllable factors and associated levels

Factor	Description	Level 1	Level 2	Level 3
P	SCG treatment	DI water rinsing (DI)	BDI	DS
C	Cd(II) concentration (mg/L)	2.5	5	10
D	SCG dose (g/L)	1	2	3
t	Contact time (min)	60	120	180
T	Temperature (℃)	25	40	55

Table 2
Design of experiments

Test identification	Factor				
	P	*C*	*D*	*t*	*T*
Test 1	1	1	1	1	1
Test 2	1	2	2	2	2
Test 3	1	3	3	3	3
Test 4	2	1	1	2	2
Test 5	2	2	2	3	3
Test 6	2	3	3	1	1
Test 7	3	1	2	1	3
Test 8	3	2	3	2	1
Test 9	3	3	1	3	2
Test 10	1	1	3	3	2
Test 11	1	2	1	1	3
Test 12	1	3	2	2	1
Test 13	2	1	2	3	1
Test 14	2	2	3	1	2
Test 15	2	3	1	2	3
Test 16	3	1	3	2	3
Test 17	3	2	1	3	1
Test 18	3	3	2	1	2

In Taguchi method, the quality characteristics are categorized into three types: larger-the-better, nominal-the-best, and smaller-the-better. As the goal of this study was to remove Cd(II) by SGC, the quality characteristic selected was larger-the-better of Cd(II) removal defined by Eq. (1). S/N for the larger-the-better quality characteristic was given by Eq. (2).

$$\text{Removal }(\%) = \left(\frac{C_0 - C_e}{C_0}\right) \times 100 \quad (1)$$

where C_0 and C_e were the initial and equilibrium concentrations of Cd(II) (mg/L), respectively.

$$S/N_{LB} = -10 \log \frac{\sum_{i=1}^{n} \frac{1}{y_i^2}}{n} \quad (2)$$

where the subscript LB represented "larger-the-better", n was the number of repetitions under the same experimental conditions, and y_i represented the measurement results.

Batch experiments were performed with the adsorbent–adsorbate suspension in a 250-mL sealed conical flask at 150 rpm and pH of 5.0 ± 0.1 in a mechanical shaking incubator equipped with a temperature controller (SB-7D Model, Deng-Yng Co.). Either NaOH or HNO_3 was used to control pH to avoid sediments of metal hydroxides. The flask contained 200 mL of Cd(II) solution. Each experiment was repeated three times. Prior to measuring Cd(II), water samples were filtered through a filter paper of cellulose acetate. Cd(II) was measured by an atomic adsorption spectrometry (Hitachi, Z-8200).

3. Results and discussion

3.1. Taguchi optimization

The results of the 18 experiments were shown in Table 3 in terms of removal average. The removal average was the average of three repeated experiments for which the individual removal was also listed in the table. The S/N ratio was calculated by Eq. (2). The results showed that the removal average of Cd(II) varied from 38.5 to 99.8%, depending on the combination of the controllable factors. From these removal averages, the related S/N ratio for each level of every individual controllable factor was drawn in Fig. 1, which illustrated the essence of Taguchi method. Fig. 1 showed that the largest S/N variation occurred with Cd(II) concentration, followed by SCG dose, SCG treatment procedure, temperature, and contact time. In other words, the most significant controllable factor was Cd(II) concentration while the least significant one was contact time.

The significance of controllable factors could be further illustrated by the range of S/N ratio (maximum S/N minuses minimum S/N) given in Table 4. A larger range implied a more significant factor and should be utilized first. The range in descending order was $C > D > P > T > t$. It could be further observed that there was no significant difference in S/N ratios for t2 and t3. This trend was further checked by analysis of variance (ANOVA) statistical approach using SPSS 17.0. The results of ANOVA showed the same order of influence on the Cd(II) removal as illustrated in Table 4 by sum of square of individual controllable factor (SSF) and percentage contribution of each controllable factor (ρF). Namely, the highest contribution was Cd(II) concentration, followed by SCG dose, SCG treatment procedure, temperature, and contact time. Hence, the optimum operating condition for the present study was $C1$, $D3$, $P3$, $T1$, and $t2$, namely Cd (II) concentration of 2.5 mg/L, SCG dose of 3 g, SCG treatment by solar energy degreasing, temperature of 25˚C, and contact time of 120 min.

3.2. Adsorption isotherms

The relationship between the amount of substance adsorbed per unit mass of adsorbent at constant temperature and its concentration in equilibrium is called

Table 3
Removal of Cd(II) and S/N ratio

Tests	Factor					Removal (%)			Average removal (%)	STD	S/N ratio
	P	C	D	t	T	$R1$	$R2$	$R3$			
Test 1	DI	2.5	1	60	25	82.5	82.4	82.3	82.4	0.08	38.32
Test 2	DI	5	2	120	40	66.9	67.6	65.8	66.8	0.74	36.49
Test 3	DI	10	3	180	55	55.4	54.3	56.2	55.3	0.78	34.85
Test 4	BDI	2.5	1	120	40	81.0	80.5	82.0	81.2	0.62	38.19
Test 5	BDI	5	2	180	55	72.3	72.8	73.2	72.8	0.37	37.24
Test 6	BDI	10	3	60	25	60.9	61.2	61.7	61.3	0.33	35.74
Test 7	DS	2.5	2	60	55	98.9	99.2	99.1	99.1	0.12	39.92
Test 8	DS	5	3	120	25	93.7	94.1	93.8	93.9	0.17	39.45
Test 9	DS	10	1	180	40	55.2	54.8	55.2	55.1	0.19	34.82
Test 10	DI	2.5	3	180	40	94.5	93.8	94.5	94.3	0.33	39.49
Test 11	DI	5	1	60	55	38.6	39.2	37.8	38.5	0.57	31.71
Test 12	DI	10	2	120	25	55.6	56.1	57.2	56.3	0.67	35.01
Test 13	BDI	2.5	2	180	25	96.8	95.6	96.4	96.3	0.50	39.67
Test 14	BDI	5	3	60	40	81.2	80.1	80.8	80.7	0.45	38.14
Test 15	BDI	10	1	120	55	40.1	41.2	41.8	41.0	0.70	32.26
Test 16	DS	2.5	3	120	55	99.8	99.8	99.7	99.8	0.05	39.98
Test 17	DS	5	1	180	25	58.6	59.2	60.4	59.4	0.75	35.47
Test 18	DS	10	2	60	40	56.8	57.2	57.0	57.0	0.16	35.12

Note: STD: Standard deviation.

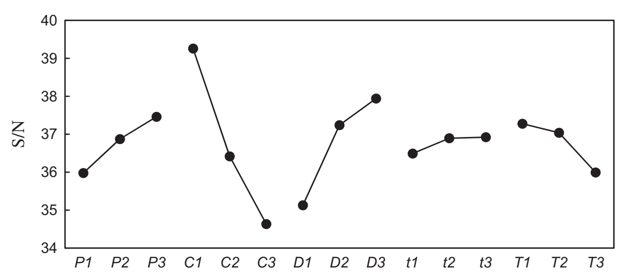

Fig. 1. Response distributions of S/N ratios.

Table 4
Response table of S/N ratios and contribution of each controllable factor

	P	C	D	t	T
Level 1	35.98	39.26	35.13	36.49	37.28
Level 2	36.87	36.42	37.24	36.90	37.04
Level 3	37.46	34.63	37.94	36.92	35.99
Range	1.48	4.63	2.81	0.43	1.29
SSF	1,254.61	13,122.07	4,308.62	104.27	479.53
ρF (%)	6.51	68.10	22.36	0.54	2.49
Rank	3	1	2	5	4
Significance	Yes	Yes	Yes	No	Yes

the adsorption isotherm. Generally, adsorption isotherms provide vital information in optimizing the use of adsorbents. Langmuir and Freundlich models are commonly used to describe the adsorption isotherms, and provide significant parameters for predicting adsorption capacities [21]. In this study, for evaluating the adsorption isotherm of various SCG treatment procedures, the combined condition with Cd(II) of 10 mg/L, contact time of 120 min, and temperature at 25˚C was adopted while the SCG dose was varied from 1 to 6 g.

3.2.1. Langmuir isotherm

The theoretical Langmuir adsorption isotherm is based on three well-known assumptions, namely adsorption cannot proceed beyond monolayer coverage, all surface sites are equivalent, and at most one adsorbed atom can be accommodated and the ability of a molecule to adsorb at a given site is independent of the occupied neighboring sites. The Langmuir equation, Eq. (3), is probably the best known and most widely applied isotherm.

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (3)$$

Its linear form can be represented by Eq. (4):

$$\frac{1}{q_e} = \frac{1}{q_m K_L}\frac{1}{C_e} + \frac{1}{q_m} \quad (4)$$

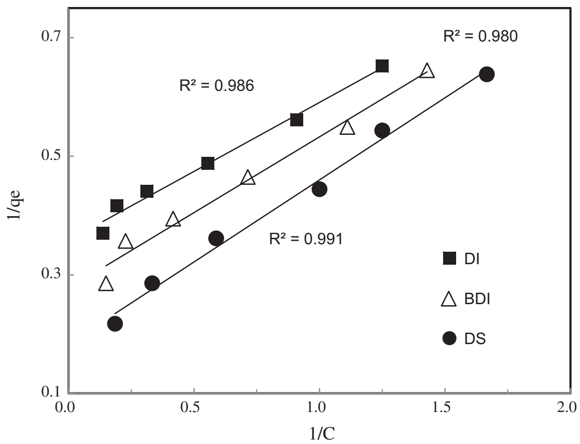

Fig. 2. Langmuir adsorption isotherms.

where q_e is the solid phase equilibrium amount of Cd(II) adsorbed per unit weight of SCG (mg/g), C_e is the equilibrium concentration of Cd(II) in the solution (mg/L), q_m is a constant related to the area occupied by the monolayer of the adsorbate which reflects the maximum adsorption capacity of Cd(II) (mg/g), and K_L (Langmuir constant) is a direct measure of the adsorption intensity (L/mg). From the plot of $1/q_e$ vs. $1/C_e$, K_L, and q_m can be determined from the slope and intercept of the linear curve. Fig. 2 showed the adsorption isotherms of three SCGs. From this plot, the deduced values of q_m were 2.79, 3.62, and 5.46 mg/g for DI, BDI, and DS treatment procedures, respectively as in Table 5. Table 5 also showed the three isotherm adsorption equations of SCG. From Table 5, the order of q_m was DS > BDI > DI, indicating that SCG degreased by solar energy had a better adsorption capacity of Cd(II).

Moreover, the characteristic parameter referred to as the separation factor "R_L" expressed in Eq. (5) [14,22] can be used to predict the affinity between the adsorbate and adsorbent.

$$R_L = \frac{1}{1 + K_L C_0} \tag{5}$$

The separation parameter R_L provides important information about the nature of adsorption. That is, irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$), or unfavorable ($R_L > 1$). The values of R_L obtained were 0.061, 0.085, and 0.132 for DI, BDI, and DS of SCG, respectively. They were all in the range of 0–1, illustrating that the three SCGs exhibited favorable adsorption. Moreover, the order of affinity between SCG and Cd(II) was DS > BDI > DI.

3.2.2. Freundlich isotherm

The Freundlich isotherm given in Eq. (6) is also well known as it is the earliest established relationship for the adsorption process. The model can be applied to adsorption on heterogeneous surfaces with the interaction between adsorbed molecules. The application of Freundlich equation also suggests that the adsorption energy decreases exponentially on completion of the adsorption centers of an adsorbent.

$$q_e = K_F C_e^{1/n} \tag{6}$$

This equation can be linearized as:

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \tag{7}$$

In these equations, the values of n and K_F were calculated from the slope and intercept of the plot of log q_e vs. log C_e. The Freundlich constant K_F indicates the adsorption capacity of the adsorbent and n is a measure of the deviation of the model from linearity of the adsorption. The adsorption is chemical, physical, and linear for n being less than, greater than, and equal to one, respectively [23]. From the results

Table 5
Langmuir and Freundlich isotherm parameters for Cd(II) adsorption

	Langmuir isotherm					Freundlich isotherm			
SCG type	q_m	K_L	R_L	R^2	Equation	K_F	n	R^2	Equation
DI	2.79	1.54	0.061	0.986	$q_e = \frac{4.30C_e}{1 + 1.54C_e}$	1.70	4.31	0.965	$q_e = 1.70C_e^{0.232}$
BDI	3.62	1.08	0.085	0.980	$q_e = \frac{3.91C_e}{1 + 1.08C_e}$	1.84	3.03	0.973	$q_e = 1.84C_e^{0.331}$
DS	5.46	0.66	0.132	0.991	$q_e = \frac{3.70C_e}{1 + 0.70C_e}$	2.09	2.10	0.989	$q_e = 2.09C_e^{0.477}$

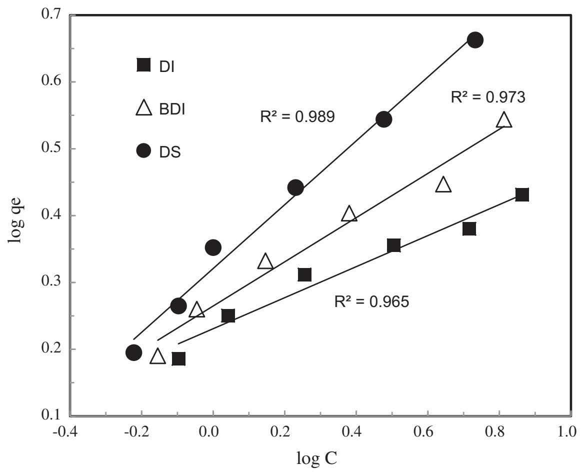

Fig. 3. Freundlich adsorption isotherms.

illustrated in Fig. 3 and Table 5, the values of n of the three SCGs were all larger than one, i.e. physical adsorption.

A comparison of adsorption of Cd(II) by various agricultural adsorbents given in Table 6 showed that different agricultural materials had different q_m. The present q_m by solar energy-treated SCG was 5.46 mg/g, similar to that of Kaikake et al. [3] by degreased coffee beans of 6.72 mg/g and Semerjian [1] by untreated Pinus halepensis sawdust of 5.36 mg/g. Our q_m was lower than most of the other agricultural adsorbents. However, we did not use any acid, alkali, solvent (which may be harmful to health and environments), or high temperature (which consumed energy) to activate the agricultural adsorbents. Hence, the application of solar energy treated SCG is both economic and environmental friendly without creating any other adverse effects.

Moreover, Table 7 summarizes the studies of using coffee as the adsorbent for further illustration. The results showed that coffee as an adsorbent could be used in many ways, such as degreased coffee bean, activated SCG, and activated coffee husk, while the adsorbate could be dyes, pesticide, methylene, and

Table 6
Comparison of q_m of Cd(II) by various agricultural adsorbents

Adsorbents	Adsorbent preparation	q_m (mg/g)	References
Esterified spent grain	Esterified by citric acid at 140˚C for 2 h (catalyst: $NaH_2PO_2 \cdot H_2O$)	473.93	[24]
Sulfurized activated nut shells	Flushed by SO_2 gas for 4 h	142.86	[25]
Protonated grapefruit peels	0.1 M HCl, 120 rpm for 6 h	Native: 95.5 protonated: 123.6	[26]
Chemically modified apple pomace	Functionalized with succinic anhydride	Native: 4.45 functionalized: 91.74	[27]
Olive cake	Dried in atmosphere for 1 year, then hot DI water washing followed by cooled water washing	65.4	[28]
Activated palm kernel shell	Activated by 1,000˚C steam	53.13	[29]
Orange waste	Tap water washing, dried at 50–60˚C	48.3	[30]
Grapefruit peels	Dried at 50–60˚C for 24 h	42.09	[31]
Wood apple shell	Treated by H_2SO_4 for 1 h	27.64	[2]
Corn stalk	Treated by NaCl at 75˚C for 2 h, then extracted with KOH 10 h followed by washing with ethanol	21.37	[32]
Untreated coffee grounds	Dried in ambient air	15.65	[33]
Bamboo charcoal	Bamboo was boiled in DI water for 1 h, then dry at 105˚C for 24 h	12.08	[34]
Ground wheat stems	Esterified by methanol	11.6	[35]
Degreased coffee beans	Soxhelt extraction by ethanol for 24 h	6.72	[3]
Present study	Degreased by solar energy	5.46	
Untreated Pinus halepensis sawdust	105˚C for 48 h	5.36	[1]
Areca (food waste)	Immersed in NaOH for 12 h, then immersed in H_2SO_4 for 12 h	1.12	[36]

Table 7
Comparison of adsorption various matter from coffee

Coffee type	Coffee preparation	Adsorbate	q_m (mg/g)	References
Coffee ground	Activated by H_3PO_4 (450˚C)	Dyes	Methylene blue: 370.4; nylosan red: 367	[12]
Activated coffee residues	Activated by $ZnCl_2$ in N_2 (600˚C, 4 h)	Formaldehyde	245	[10]
Activated coffee husk	Carbonization at 673 K in N_2 (2 h)	Dye(RBO3R)	66.76	[37]
Degreased coffee bean	Degreased by NaOH	Malachite Green	55.3	[14]
Untreated coffee husks	DI water washing	Methylene	111	[38]
Degreased coffee beans	Soxhelt extraction by ethanol 24 h	Cd(II)	6.72	[3]
Exhausted coffee ground	Leached by NaOH solution	Heavy metals	Cu(II): 3.1 Pb(II): 15.8 Zn(II): 6.1 Cd(II): 3.2	[9]
SCG	Degreased by solar energy	Cd(II)	5.46	Present study
Activated SCG	Chemical activation agents: H_3PO_4 and $ZnCl_2$	Pesticide (malathion)	1.168	[13]

heavy metals. Our result of Cd(II) adsorption was better than that of Utomo and Hunter [9]. They used NaOH solution to leach coffee ground and the values of q_m for Cu(II) and Cd(II) were 3.1 and 3.2 mg/g, respectively. In addition, q_m of Cd(II) adsorption of Kaikake et al. [3] using ethanol-degreased coffee beans was 6.72 mg/g, similar to ours. Overall observation revealed that that coffee material was an effective adsorbent. It could adsorb not only dyes and pesticide but also heavy metals.

4. Conclusions

This study evaluated the feasibility of employing SCG for Cd(II) adsorption using Taguchi method to determine the optimum removal condition through 18 experiments. The results showed that Cd(II) removal varied from 38.5 to 99.8%, depending on how the controllable factors were combined. The influencing extent of the controllable factors of Cd(II) removal in descending order was $C > D > P > T > t$. Namely, concentration of Cd(II) was the most influencing factor while contact time was the least. For the effect of treatment procedure of SCG, the removal efficiency of Cd(II) in descending order was DS > BDI > DI. In other words, the SCG degreased by solar energy was more effective than the other two types of SCG. The maximum adsorption capacity of Cd(II) by DI, BDI, DS was 2.79, 3.62, and 5.46 mg/g, respectively. Further, both Langmuir and Freundlich adsorption isotherms showed that SCG was an effective adsorbent for Cd(II) removal from wastewater.

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