

# Synthesize and Application of SiO<sub>2</sub>/CPW Nanocomposite for Removal of Cadmium from Industrial Wastewater

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## Abstract

The coconut peel waste (CPW) was chemically spiked with silica nanoparticles to develop a novel nanocomposite (SiO<sub>2</sub>/CPW). The new nanocomposite was characterized by FTIR, SEM and Dynamic Light Scattering method. Adsorption of Cd ions onto SiO<sub>2</sub>/CPW was studied in batch mode as a function of pH, contact time, adsorbent dosage and initial concentration and temperature. The maximum removal of Cd<sup>2+</sup> ions was at pH=6.5 and adsorbent dosage=0.1 g. The experiments showed that the adsorption process was quick and about 74.5% of total cadmium was removed within 5 min. Cadmium uptake by the new adsorbent was best described by pseudo-second order model. Using the equilibrium concentration constants obtained at different temperatures, various thermodynamic parameters have been calculated. The results indicated that Cadmium adsorption was feasible, spontaneous, and endothermic. The suggested sorbent proved the great potential in cadmium removal from water and wastewater.

## Keywords

Removal; Cadmium; Coconut peel waste (CPW); Nanocomposite; Wastewater.

## 1. INTRODUCTION

One of the major threats of heavy metals on human health is contact with contaminated water due to industrial development and population growth [1-3]. Generally, heavy metals cannot be degraded or eliminated over time and are dangerous even in ultratrace quantities. In the field of water rule, cadmium is on the list of high-priority pollutants [4]. Cadmium is toxic transition metal that affects the environment through anthropogenic and/or natural processes [5]. It is considered one of the most hazardous metals. It is used in many industries, including batteries, paint, and dyes, but municipal waste is the most complex source due to its nearness to human reach [6]. Cadmium can be exist in the industries as cadmium nitrate, cadmium sulfate and it is more likely to leach from industrial wastes and is the most toxic to living organisms [7,8]. Cadmium has been included in the list of priority pollutants to control EPA priority due to the persistent Cadmium and toxicological properties. In drinking water and industrial wastewater, the permissible concentration has been set at 0.005 mg/L by US Environmental Protection Agency (USA-

EPA), while the World Health Organization has set it at level 0.003 mg/L [9].

Previous studies have reported that the food chain is the main route of cadmium ingested by humans and that it easily accumulates in living organisms due to its nondegradable nature [10,11]. Thus, the removal of cadmium from water and wastewater is an important subject and reducing environmental pollution and its harmful effects on humans should be seriously considered.

Several conventional methods have been used for Cd removal from water and wastewater. The adsorption technique is widely considered as an efficient method for eliminating toxic metals due to its simple operation, economical, high efficiency, and low cost compared with traditional methods such as ion exchange, ultrafiltration, precipitation, membrane and electrochemical processes [12-18]. Different adsorbents performed to evaluate the adsorption and removal toxic metals from aqueous solutions [19-22]. However, so far, no reports of the adsorption of heavy metals using coconut peel waste (CPW) was chemically spiked with silica nanoparticles has been conducted.

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In this study, a low cost nanoadsorbent is developed by chemically modified coconut peel waste (CPW), and evaluated its potential for the removal of Cd<sup>2+</sup> ions from water and wastewater. Nano silica has been used as a coating material to explore the surface functionality and further enhancing adsorption performance of coconut peel waste and finally the proposed adsorbent was applied for purification of wastewater containing cadmium ions.

## 2. EXPERIMENTAL

### 2.1. Materials and methods

The starting solutions were tetraethylorthosilicate (TEOS) (99.9%, Aldrich Chemical Co.), ethanol (99.9%, Sigma Chemical Co). Cd(NO<sub>3</sub>)<sub>2</sub> (99% wt) and all other chemicals and reagents used in this study were purchased from Merck and were of analytical grade without further purification. All solutions were prepared using double distilled water.

### 2.2. Preparation of SiO<sub>2</sub> nanoparticles

SiO<sub>2</sub> nanoparticles were prepared by Stobber method [23]. 5 mL tetraethoxysilane was added to the ethanol-water 2:1 (v/v) mixture under sonication. Then, 20 mL of 25% ammonia was dropped slowly into the mixture. The obtained white gel was centrifuged, washed with deionized water and dried in oven at 100 °C for 4h. Finally, fine white nanopowders of SiO<sub>2</sub> were obtained.

### 2.3. Preparation of dried biomass of coconut peel waste

Coconut peels were collected from its fruit as waste after the consumption of coconut. They washed with double distilled water and dried in 25 °C. After that, they were crushed, ground and sieved through a 100-mesh sieve. They washed with distilled water and dried in oven at 100 °C for 24 h. The obtained powder was stored in plastic container for preparation of the nanocomposite.

### 2.4. Preparation of silicated Coconut peel waste (SiO<sub>2</sub>@CPW) nanocomposite

3.0 g of the prepared SiO<sub>2</sub> nanoparticles were dispersed in ethanol-water (1:1) mixture at room temperature. Then, 15 g of coconut peel powder was added to the above reaction mixture. The suspension was stirred under continuous stirring for 90 min. The final product SiO<sub>2</sub>@CPW was collected by centrifugation and then washed twice with double distilled water/ethanol mixture. Finally, the particles were dried in oven at 100 °C for one night.

### 2.5. Characterization

The functional groups of nanosilica and SiO<sub>2</sub>@CPW nanocomposite were identified by FT-IR spectra (in KBr pellet) in the range of 460–

4000 cm<sup>-1</sup>. The microstructure of the prepared nanocomposite was observed by Scanning Electron Microscopy (SEM) (Cam Scan MV2300). The Dynamic Light Scattering (DLS) test was also employed to specify the average particle size and distribution of the synthesized nanocomposite.

### 2.6. Adsorption procedure

Batch adsorption experiments were carried out by shaking 0.1g of the adsorbent (SiO<sub>2</sub>@CPW) with known volume of the Cd<sup>2+</sup> aqueous solutions in stoppered pyrex glass flask, at a fixed temperature. The pH of the solutions was adjusted with HNO<sub>3</sub> or NaOH 0.1 M solution and the shaking speed was 200 rpm for 5 min. At the end of the adsorption period, the sorbent was separated from the suspension and the cadmium concentration was determined using FAAS.

The amount of Cd(II) ions adsorbed and percentage of removal of Cd(II) ions (Re%) was calculated using Equations (1) and (2).

$$q_e = V(C_0 - C_e)/M \quad (1)$$

$$\text{Re\%} = [(C_0 - C_e)/C_0] \times 100 \quad (2)$$

That  $q_e$  is the amount of adsorbed Cd(II) ion per gram of the adsorbent,  $C_0$  is initial concentration and  $C_e$  is the equilibrium concentration of the Cd(II) (mg L<sup>-1</sup>) that is obtained from calibration curve and  $M$  is the mass of nanocomposite (g).

## 3. RESULTS AND DISCUSSION

### 3.1 Characterization of the adsorbent

SiO<sub>2</sub>@CPW nanocomposite was characterized employing various techniques including FT-IR SEM and DLS. FT-IR spectra of nanosilica and SiO<sub>2</sub>@CPW nanocomposite is shown in Fig. 1. The FT-IR peaks between 466-1100 cm<sup>-1</sup> is related to the vibration of the Si-O-Si band, in which the peak at 466 cm<sup>-1</sup> is assigned to the flexural vibration of Si-O-Si, while the peaks at 801 and 954 cm<sup>-1</sup> are ascribed to symmetric stretch of Si-O-Si and 1133 cm<sup>-1</sup> is related to the asymmetric stretch of Si-O-Si (Fig. 1b). The typical absorption band at 3200–3488 cm<sup>-1</sup> in the FT-IR spectrum of nanocomposite may be attributed to -OH groups of carbohydrates, pectin and lignin. The absorption peak at 2894 cm<sup>-1</sup> is corresponds to C-H symmetric and asymmetric vibrations and peaks at 1739 cm<sup>-1</sup> is attributed to carboxylic groups (COOH and COOCH<sub>3</sub>) (Fig. 1a).

SEM analysis was done to identify the surface and morphology of SiO<sub>2</sub>@CPW nanocomposite. Fig. 2 shows aggregate silica nanoparticles on the surface of CPW. Particle size and distribution of the synthesized nanocomposite was evaluated by Dynamic light scattering (DLS). The DLS pattern

(Fig. 3) showed that the synthesized nanoparticles had an average diameter about 10 nm.

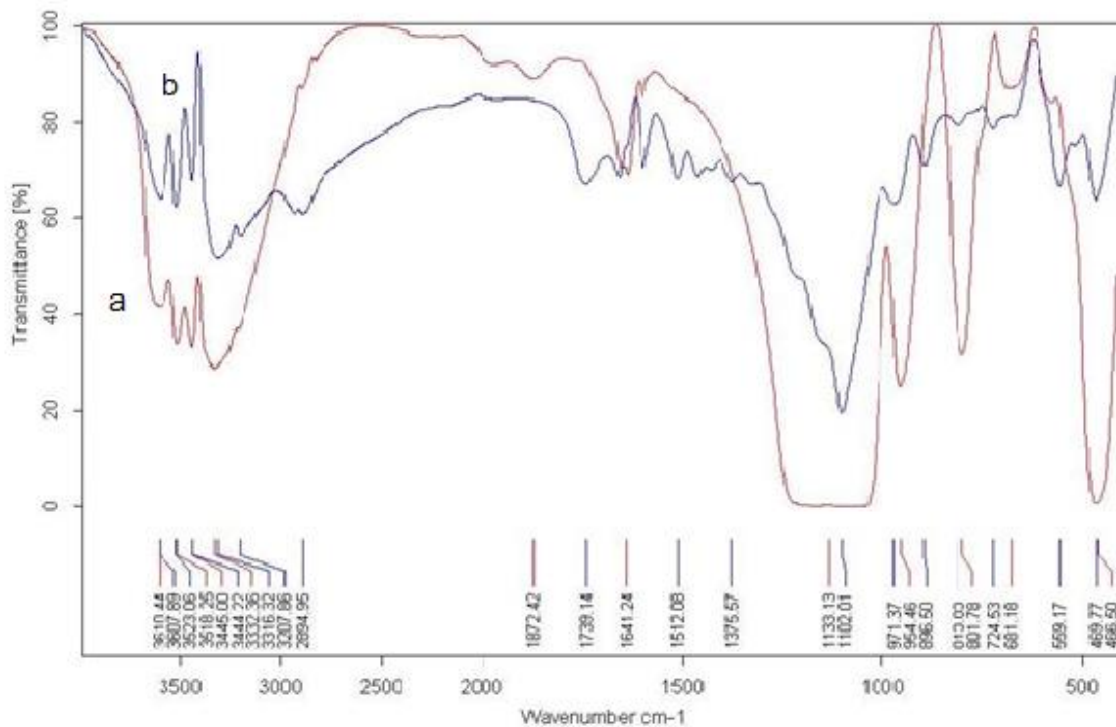


Fig. 1. (a) nanosilica and (b) FTIR spectra of SiO<sub>2</sub>@CPW nanocomposite.

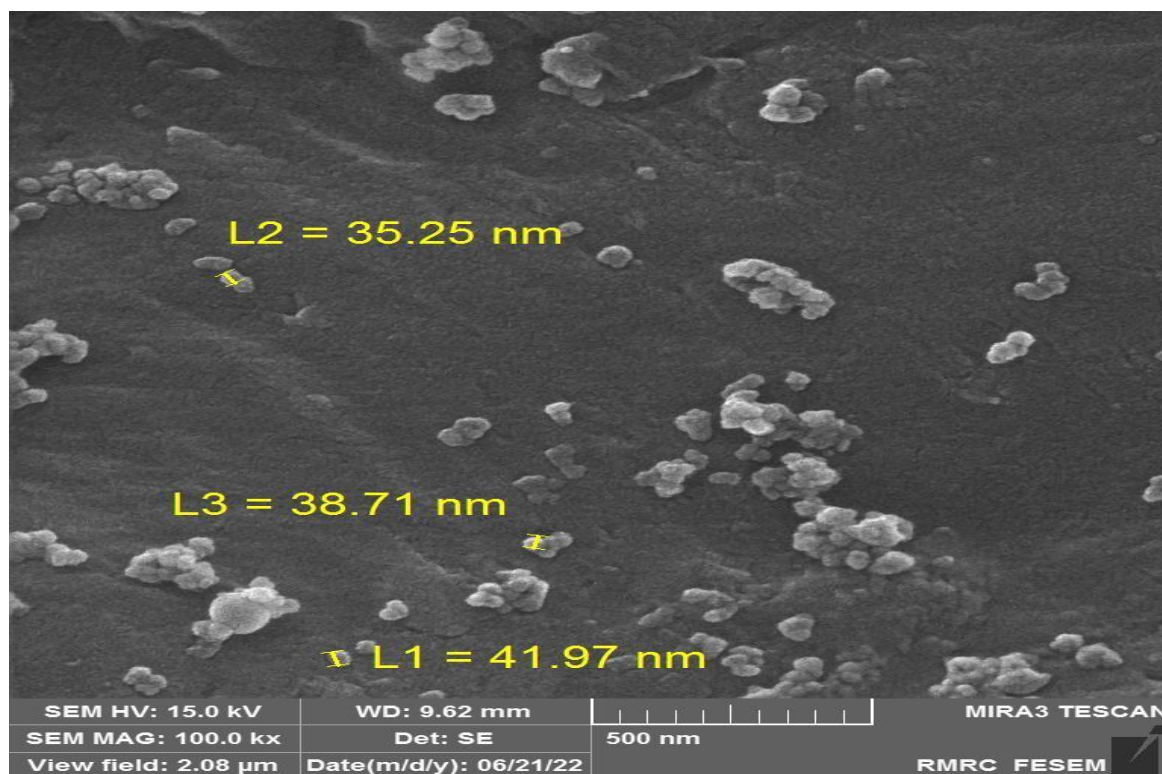


Fig. 2. SEM image of SiO<sub>2</sub>@CPW nanocomposite.

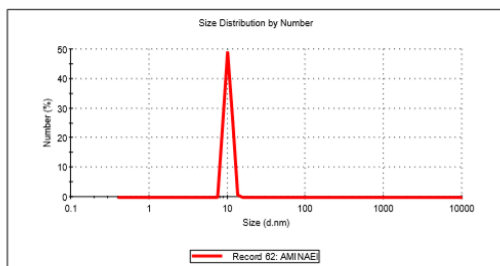


Fig. 3. DLS spectrum of nanocomposite.

### 3.2. Effect of pH

The affinity of adsorbent functional sites for cadmium ions depends on the initial pH of the solution. The effect of pH was studied in a pH range of 2.0–8.5. The sorption capacity and the removal percentage increased with increasing pH and then gradually decreased (Fig. 4).

In acidic solutions, high  $H^+$  ions concentration is being attributed to competitive with  $Cd^{2+}$  for occupying active sites of  $SiO_2@CPW$ . Also, functional groups on the adsorbent protonated and caused electrostatic repulsion to  $Cd^{2+}$  ions. With increasing pH, electrostatic attractions occur between cationic  $Cd^{2+}$  ions and anionic adsorbent surface. Since at  $pH > 7.5$ , Precipitation occurs due to forming cadmium hydroxide, the experiments were performed at pH 6.5.

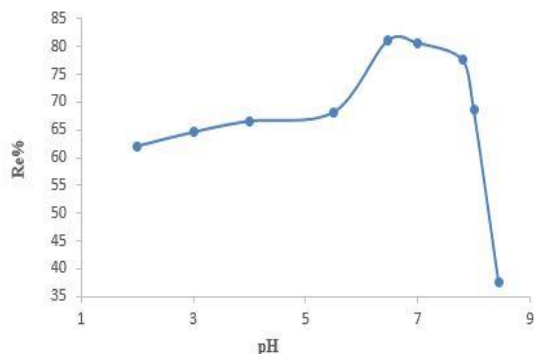


Fig. 4. Effect of pH on the removal of cadmium.

### 3.3. Effect of sorbent dose

The influence of sorbent dose on cadmium removal was studied by varying the sorbent dose from 0.01 to 0.15 g. The percent removal of cadmium increased from 68.3% to 79.2% with an increase in adsorbent dose (Fig.5). The removal of cadmium at  $m > 0.1$  g is almost constant. An increase in the removal with the adsorbent dosage can be attributed to greater surface area and the availability of more adsorption sites. At  $m < 0.1$  g, the adsorbent surface becomes saturated with cadmium and the residual concentration in the solution is large. Thus, the dose of the adsorbent was selected 0.1 g for the subsequent experiment.

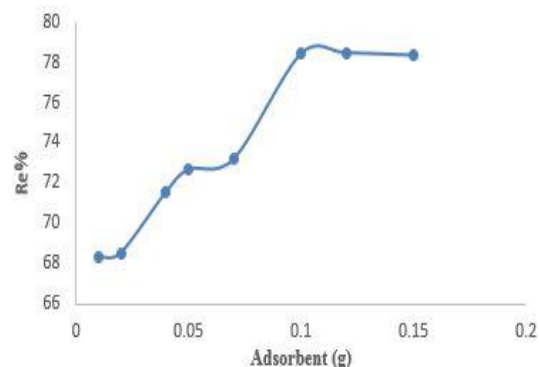


Fig. 5. Effect of adsorbent dosage on the removal of cadmium.

### 3.4. Effect of contact time and initial concentration

Effect of contact time for removal of cadmium was examined. The experimental were carried out in the time range 5 to 90 min at room temperature. The examination of time showed that cadmium adsorption onto new nanocomposite was very quick, as in the first minutes, high removal efficiency was obtained and then almost remained constant (Fig. 6). 5 min was selected as equilibrium time for more experiments.

The effect of cadmium concentration on the sorption  $SiO_2@CPW$  by nanocomposite was investigated with different concentration ( $5-25 \text{ mg.L}^{-1}$ ) of cadmium. With increasing concentration of the solution,  $q_e$  increased, while  $Re\%$  decreased (Figs 7, 8). At low initial concentration, the surface area and the availability of adsorption sites were high, and the cadmium ions easily adsorbed and removed. At high initial concentration, the total available sites are limited, that resulting in a decrease in removal of the cadmium. The increased  $q_e$  at high concentration can be attributed to enhanced driving force.

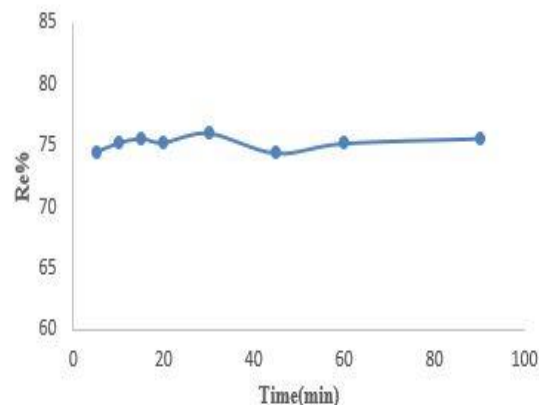
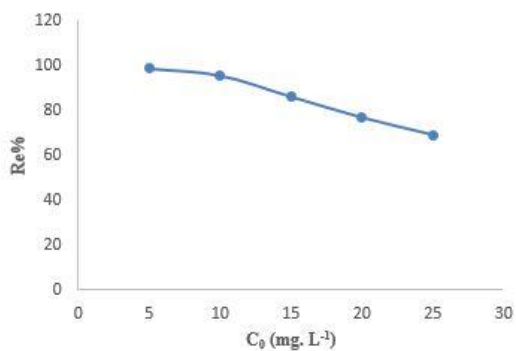
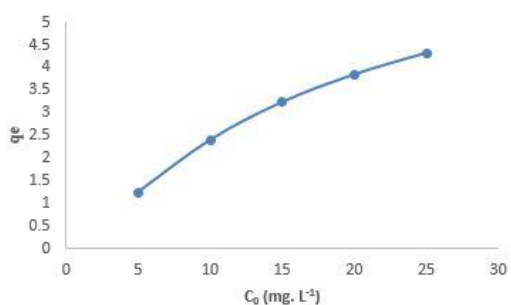


Fig. 6. Effect of contact time on cadmium adsorption.



**Fig. 7.** Effect of concentration on the removal of cadmium.



**Fig. 8.** Effect of concentration on the q<sub>e</sub>.

### 3.5. Effect of temperature on cadmium adsorption

Effect of temperature and thermodynamic parameters were examined, also. Study of the temperature dependence of adsorption gave valuable knowledge about the enthalpy and entropy changes during adsorption. The removal of cadmium on the SiO<sub>2</sub>@CPW was examined at 284, 292, 297 and 311 K to determine the adsorption thermodynamic parameters. Adsorption ability of the SiO<sub>2</sub>@CPW increased with increasing temperature, illustrating that adsorption of cadmium on the SiO<sub>2</sub>@CPW is an endothermic process.

The Gibbs free energy change,  $\Delta G^0$ , is fundamental criterion of spontaneity. The sorption process of cadmium can be summarized by the following reversible process. The equilibrium constant ( $K_c$ ) is defined as:

$$K_c = (C_0 - C_e) / C_e \quad (3)$$

where  $C_e$  is the concentration of cadmium at equilibrium. The  $K_c$  value is used in the following equation to determine the  $\Delta G^0$  of sorption.

$$\Delta G^0 = -RT \ln K_c \quad (4)$$

The enthalpy ( $\Delta H^0$ ) and entropy ( $\Delta S^0$ ) can be calculated from the slope and intercept of Vant Hoff equation of  $\Delta G^0$  versus T.  $\Delta G^0 = \Delta H^0 - T \Delta S^0$

R the gas constant (8.314 J mol<sup>-1</sup>K<sup>-1</sup>) and T is the absolute temperature (K). The obtained  $\Delta G^0$  values and the adsorption thermodynamic parameters are given in Table 1. The positive value  $\Delta S^0$  shows the

increase in degree of freedom or increase the disorder of adsorption cadmium on the sorbent and the positive value  $\Delta H^0$  indicates that adsorption process is endothermic.

**Table 1.** Thermodynamic Parameters for adsorption of Cd.

T (K)	$\Delta G^0$ (J mol <sup>-1</sup> )	$\Delta S^0$ (J mol <sup>-1</sup> K <sup>-1</sup> )	$\Delta H^0$ (kJ mol <sup>-1</sup> )
284	-2478.35	34.54	7.289
292	-2724.07		
297	-3144.41		
311	-3388.21		

### 3.6. Equilibrium adsorption isotherms

The adsorption isotherms of cadmium on SiO<sub>2</sub>@CPW were examined in the concentration range 5.0-25.0 mg·L<sup>-1</sup> using Langmuir and Freundlich models to understand the adsorption mechanism. Linear forms of Langmuir and Freundlich isotherms are given in Eqs. (6) and (7).

$$C_e/q_e = (1/Qb) + (1/Q) C_e \quad (6)$$

$$\log q_e = \log K_f + 1/n \log C_e \quad (7)$$

$q_e$  is the amount of adsorbed Cd per gram of the adsorbent,  $b$  is Langmuir constant and  $Q$  is adsorption capacity expressed in mg g<sup>-1</sup>,  $n$  is the Freundlich constant, and  $K_f$  is the adsorption coefficient. The results showed that the slopes of both plots were high ( $R^2=0.99$  for Langmuir and  $R^2=0.98$  for Freundlich), thus adsorption of cadmium on the new nanocomposite fit for both models Langmuir and Freundlich isotherm.

### 3.7. Adsorption kinetics

The adsorption kinetics described by the relationship between cadmium uptake and contact time that is discussed by Pseudo-First order and Pseudo-Second order, Eqs. (8, 9).

The Pseudo-First order equation is:

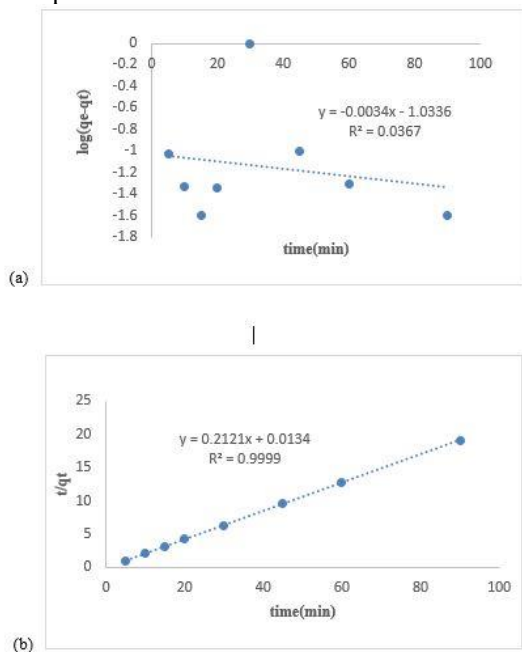
$$\log(q_e - q_t) = \log q_e - (k_1 / 2.303) t \quad (8)$$

and the Pseudo-Second order equation is:

$$(t/q_t) = (1/k_2 \cdot q_e^2) + t/q_e \quad (9)$$

where  $q_t$  is the amount of cadmium adsorbed at 't' time (mg/g),  $t$  is the contact time (min),  $q_e$  is the amount of cadmium adsorbed at equilibrium (mg/g),  $k_1$  is the rate constant of Pseudo-First order reaction (min<sup>-1</sup>) and  $k_2$  is the rate constant of Pseudo-Second-order reaction (g mg<sup>-1</sup> min<sup>-1</sup>). The slope and intercept of plot of  $\log(q_e - q_t)$  against time (min) were used to determine the First order rate constant  $k_1$ . The calculated parameters of  $R^2$ ,  $q_{e,cal}$  and  $q_{e,exp}$  were listed in Table 2. It was observed that correlation coefficient ( $R^2$ ) was low and experimental data are not fitted well for Pseudo-First order reaction. Hence, the adsorption mechanism cannot be well described by Pseudo-First order kinetics. The Pseudo-Second order rate

constant  $k_2$  was calculated from the slope and intercept of the plot  $t/q_t$  against time (Fig. 9). The calculated  $q_e$  ( $q_{e,cal}$ ) was in good agreement with experimental  $q_e$  ( $q_{e,exp}$ ) with correlation coefficient high (Table 2). These results confirm the well fitting of Pseudo-Second order model for adsorption of cadmium on to the sorbent.



**Fig. 9.** (a) Pseudo-First order and (b) Pseudo-Second order plot for cadmium adsorption.

**Table 2.** Adsorption kinetic constants for adsorption of Cd on the  $\text{SiO}_2@\text{CPW}$

First-order kinetic model				Second-order kinetic model		
$q_{exp}$	$q_{cal}$	$K_1$	$R^2$	$q_{cal}$	$K_2$	$R^2$
( $\text{mg.g}^{-1}$ )	( $\text{mg.g}^{-1}$ )	( $\text{L.min}^{-1}$ )		( $\text{mg.g}^{-1}$ )	( $\text{L.min}^{-1}$ )	
4.75	1.01	0.0078	0.037	4.72	3.36	0.999

### 3.8. Desorption

The desorption of Cd loaded on the adsorbent was tested using  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$  and  $\text{HNO}_3$  with different concentrations. Acids with concentration of 1.0 and 0.5 M were applied for this purpose. Desorption of cadmium with  $\text{HNO}_3$  1.0 M showed the highest desorption percent.

### 3.9. Real Samples

The proposed adsorbent was applied for removal of Cd on effluent samples of steel manufacture. Industrial effluent was collected from Steel Butia Iranian manufactory located near Kerman, Iran. Batch experiments were carried out under optimum conditions similar to the synthetic samples. The recoveries of the spiked samples are given in Table 3. The results showed that the

proposed adsorbent can be applied for the removal of toxic Cd from aqueous environments.

**Table 3.** Removal of cadmium from the wastewater samples.

Sample	cadmium ( $\text{mg. mL}^{-1}$ )	
	Spiked	Removal (%)
Wastewater	0.0	-----
Effluent <sup>a</sup>	5.5	86.17
	10.0	81.46
	15.0	81.58
RO <sup>b</sup>	0.0	-----
Effluent	5.5	100.00
	10.0	94.55
	15.0	72.47

<sup>a</sup> Steel Butia Iranian manufactory, Kerman

<sup>b</sup> After reverse osmose, Steel Butia Iranian manufactory, Kerman,

## 4. CONCLUSIONS

In the present paper, a novel anocomposite ( $\text{SiO}_2@\text{CPW}$ ) was synthesized, characterized and applied for removal of cadmium from wastewater. The dispersion of nanosilica onto the surface of CPW is confirmed by SEM. It was observed that the equilibrium was achieved within 5 min. The endothermic and spontaneous nature of the adsorption was confirmed by thermodynamic studies. The adsorption isotherms showed good fit for both models Langmuir and Freundlich. The main advantages of the proposed adsorbent are: low toxicity of the sorbent and environmentally friendly and green, abundantly available, low cost and easy preparation, re-usability, rapid uptake (5 min) and adsorption high efficiency of Cd from wastewater.

### Declaration of interest

There are no conflicts to declare. The authors report no conflicts of interest. Also, the authors are responsible for the writing and content of this article.

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## سنتز و کاربرد نانو کامپوزیت SiO<sub>2</sub>/CPW برای حذف یونهای کادمیم از پسابهای صنعتی

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### چکیده

پودر پوست نارگیل (CPW) بطور شیمیایی با نانوذرات سیلیس اصلاح و نانو کامپوزیت SiO<sub>2</sub>/CPW تهیه گردید. نانو کامپوزیت جدید، توسط تکنیکهای FT-IR, SEM و DLS شناسایی شد. جذب کادمیم بر روی SiO<sub>2</sub>/CPW به صورت ناپیوسته و بعنوان تابعی از pH, زمان تماس, مقدار جاذب, غلظت اولیه ی کادمیم و دما بررسی گردید. بیشترین حذف یونهای کادمیم در pH= ۶/۵ و مقدار جاذب ۰/۱ گرم بود. آزمایشات نشان داد که فرایند جذب سریع بوده و ۷۴/۵ درصد از کل کادمیم در ۵ دقیقه اول حذف میگردد. جذب کادمیم توسط جاذب جدید از معادله شبه مرتبه اول پیروی کرد. با استفاده از ثابتهای تعادلی غلظت در دماهای مختلف پارامترهای ترمودینامیکی محاسبه شدند. نتایج، دلالت بر جذب کادمیم بصورت خود به خودی و گرماگیر داشت. جاذب پیشنهادی پتانسیل خوبی در حذف کادمیم از آب و پسابهای صنعتی دارد.

### کلید واژه ها

حذف، کادمیم، پوست نارگیل، نانو کامپوزیت، پساب صنعتی.