



## Cellulose Adsorbent from Biocompatible Bamboo Chopsticks and ZnO for Removal of Chromium in Wastewater

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The daily waste material of cellulose obtained from bamboo chopsticks was combined with ZnO to prepare an optimized adsorbent (Cel-ZnO) for the removal of Cr(VI) present in wastewater. This work investigated the adsorption of Cr(VI) by Cel-ZnO in comparison to cellulose (Cel). The influence of functional groups and the amount of sorbent on adsorption was studied by Fourier transform infrared (FTIR), scanning electron microscope (SEM) and thermogravimetric analysis (TGA). Moreover, the effects of pH, concentration, time and temperature on adsorption were studied along with isotherm and kinetic studies. The optimum conditions for Cr(VI) adsorption were identified as 0.0050 g of Cel-ZnO adsorbent, 250 ppm initial concentration of Cr(VI) at a pH of 3 and adsorption time of 360 min at 30 °C. The adsorption capacity of Cel was 91.18 mg/g and Cel-ZnO had an adsorption capacity of 197.26 mg/g. It can be concluded that both Cel and Cel-ZnO have good adsorption efficiencies for Cr(VI), with the latter having an additional advantage of reusability.

**Keywords:** Bamboo chopsticks, Cellulose, Wastewater, Biocompatible, Chromium.

### INTRODUCTION

At present, the environmental problems are tremendously increasing in Thailand due to technological advancements. This has led to the expelling of many toxins and wastes from the automobile industries, chemical fertilizers and pesticides contaminated with heavy metals from agricultural domains, laboratory chemicals and hazardous wastewater from scientific labs [1]. The wastewater contaminated with such inorganic and organic substances, as well as heavy metals, are not treated before discharging into natural water sources. This unhealthy practice causes serious impacts on the environment and human health [2].

Chromium contaminants, either in Cr(III) or Cr(VI) form, in water sources cause high toxicity [3]. It is more than a hundred times more toxic than Cr(III). Once, Cr(VI) enters the human body, it will accumulate, causing chronic toxicity and cancer in various organs. On the other hand, Cr(III) helps the metabolism of fats and sugars in the human body, but if taken in large quantities, it causes harm. If animals are fed with chromium contaminated food, especially Cr(VI), even in small doses, it retards their growth and may even lead to cancer [4-6]. Therefore, it is necessary to remove chromium ions from wastewater

before releasing it into natural water sources. There are several treatment processes, such as chemical reduction [7], membrane separation [8], ion-exchange [9] and adsorption [10]. Among all the separation processes, adsorption is the most suitable one for chromium ions removal. However, the challenge is to fabricate an efficient and environmentally friendly adsorbent. The most used adsorbents for heavy metal removal are clay, silica, zeolite, activated carbon and cellulose [11,12]. It is possible to derive better adsorption performances from these adsorbent materials by improving their surfaces. The detection of bioactive substances, for ZnO nanoparticles, with high surface area and having good catalytic, non-toxic and antibacterial properties is an emerging field of study. This prompted us to study the adsorbent efficiency of waste material of cellulose obtained from bamboo chopsticks was combined with ZnO (Cel-ZnO) to prepare an optimized adsorbent.

### EXPERIMENTAL

Zinc acetate dihydrate ( $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ ), chromium (VI) oxide ( $\geq 99.99\%$ ), sodium hydroxide, zinc standard solution 1000 mg/L were purchased from Merck Co., Inc. (Germany). Cellulose fibers (Cel) were collected in the form of bamboo

chopsticks from the local market of Buriram, Thailand. All chemicals were of analytical grade and used without purification.

A thermo scientific spectrometer (Thermo Nexus 470FT-IR, Nicorette, USA) was used to obtain the Fourier transform infrared (FT-IR) spectra of different materials in the range 4000-400  $\text{cm}^{-1}$ , while a scanning electron microscope equipped (Hitachi FE-SEMS-4800) was used to determine their surface morphologies. Thermogravimetric analyses (TGA) were performed using a Thermo Scientific SDT Q600 series thermogravimetric analyzer (TA instrument).

**Synthesis of Cel-ZnO adsorbent:** Bamboo chopsticks were cut into smaller particles and spun to get fibres with even smaller particle sizes. Hemicellulose and lignin were then removed by base pre-treatment to make cellulose fibres purer. Initially, 5 g of cellulose fibre was immersed in  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  for 1 h under continuous stirring at 200 rpm. The mixture was then sonicated for 20 min in a water bath and followed by NaOH pre-treatment for 10 min to reduce zinc nanoparticles. It was then annealed for 24 h at 30 °C. Finally, at this stage, cellulose zinc oxide was obtained as the product and had been used thereafter for further Cr(VI) absorption studies [13].

### Optimized conditions

**Effect of pH:** Approximately 0.0050 g of the adsorbed sample was weighed and immersed in a 50 mL Cr(VI) solution of concentration 250 mg/L with varying pH levels from 2 to 7. It was then shaken at a speed of 150 rpm for 360 min. The mixtures were centrifuged and the resulting solutions were further analyzed.

**Effect of time:** Approximately 0.005 g of the adsorbed sample was weighed and immersed in a 50 mL Cr(VI) solution of concentration 250 mg/L at a pH level of 3 and shaken at a speed of 150 rpm for different time durations. The time durations employed for the study were 1, 3, 5, 10, 20, 30, 60, 180 and 360 min. The resultant mixture solutions were centrifuged and subjected to further analysis.

**Effect of concentration:** Approximately 0.005 g of adsorbed sample was weighed and immersed in 50 mL Cr(VI) solution of varying concentrations (10, 25, 50, 100, 150, 200, 250, 300 and 500 mg/L) at pH 3 and shaken at a speed of 150 rpm for 360 min. The resultant solutions from each mixture were centrifuged and examined for further analysis.

**Adsorption studies:** To measure the concentration of Cr(VI) solution adsorbed, the sample solution was quantified in comparison to the standard solution of potassium dichromate. A 1:5 ratio of Cr(V) to 1,5-diphenylcarbazide (DPC) solution was used to form a purple complex. The absorbance was measured using a UV-Vis spectrophotometer. At a wavelength of 540 nm, the amount of Cr(VI) absorbed (q) was calculated as follows [14-16]:

$$Q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

$$\frac{C_e}{Q_e} = \frac{1}{bQ_m} + \frac{C_e}{Q_m} \quad (2)$$

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (3)$$

$$\ln(Q_e - Q_t) = \ln Q_e - (k_1)t \quad (4)$$

$$\frac{t}{Q_t} = \frac{1}{kQ_e^2} + \frac{t}{Q_e} \quad (5)$$

where  $Q_e$  (mg/g) represents adsorption capacity,  $C_0$  and  $C_e$  represent the initial and equilibrium concentrations of Cr(VI) (mg/L), respectively,  $V$  represents the volume of the solution;  $m$  represents the weight of adsorbent;  $Q_m$  is the Langmuir monolayer sorption capacity;  $b$  (L/mg) is the Langmuir equilibrium adsorption constant;  $K_f$  and  $n$  are the Freundlich equilibrium adsorption constants;  $Q_t$  is the amount of adsorbed in mg/g at time  $t$  (min);  $k_1$  ( $\text{min}^{-1}$ ) is the rate constant of pseudo-first-order adsorption and  $k_2$  (g/mg·min) is the pseudo-second-order rate constant.

**Recyclability studies:** In this experiment, desorption of Cr(VI) from the prepared adsorbent (Cel-ZnO) was performed using deionized water as solvent washing regeneration incorporated with thermal treatment method. The spent adsorbents were dipped at 80 °C for 30 min then followed by drying at 105 °C for 10 h in an oven. The optimal conditions were maintained throughout the reusability experiments.

## RESULTS AND DISCUSSION

**FTIR studies:** The FTIR analysis of Cel and Cel-ZnO was performed and compared (Fig. 1). The cellulose showed that a broad peak appeared at 3437  $\text{cm}^{-1}$ , which was characteristic stretched oscillations of cellulose O-H bonds. Moreover, the peaks at 1730 and 1434  $\text{cm}^{-1}$  were due to the stretched oscillation peaks of the C=O bonds, indicating that the fibres contained lignin and hemicellulose. However, these peaks do not appear in the spectrum of base-treated cellulosic fibres [17]. A peak at 1639  $\text{cm}^{-1}$  appeared due to the stretched oscillation peak of the C=O after the elimination of hemicellulose upon base extraction. In Cel-ZnO adsorbent, a sharp and intense band at 540  $\text{cm}^{-1}$  indicates the existence of the Zn-O vibrations [18].

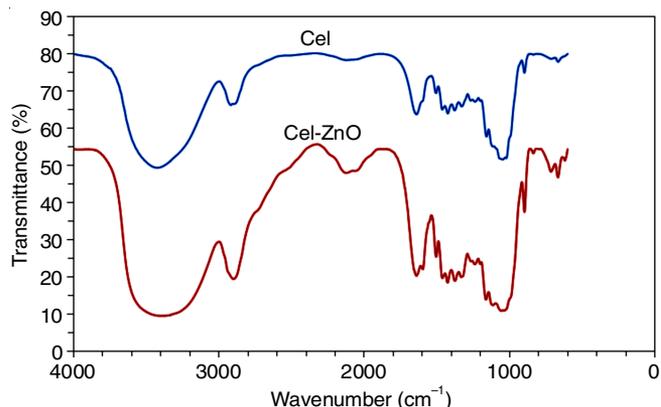


Fig. 1. FTIR spectrum of Cel and Cel-ZnO sorbents

**Morphology:** The surface area characterization of Cel, Cel-Cr, Cel-ZnO and Cel-ZnO-Cr using SEM technique is shown in Fig. 2a-d. It was found that base treatment makes the cellulose surface more suitable for adsorption and the ZnO solution makes the surface more favourable to adsorb chromium metal.

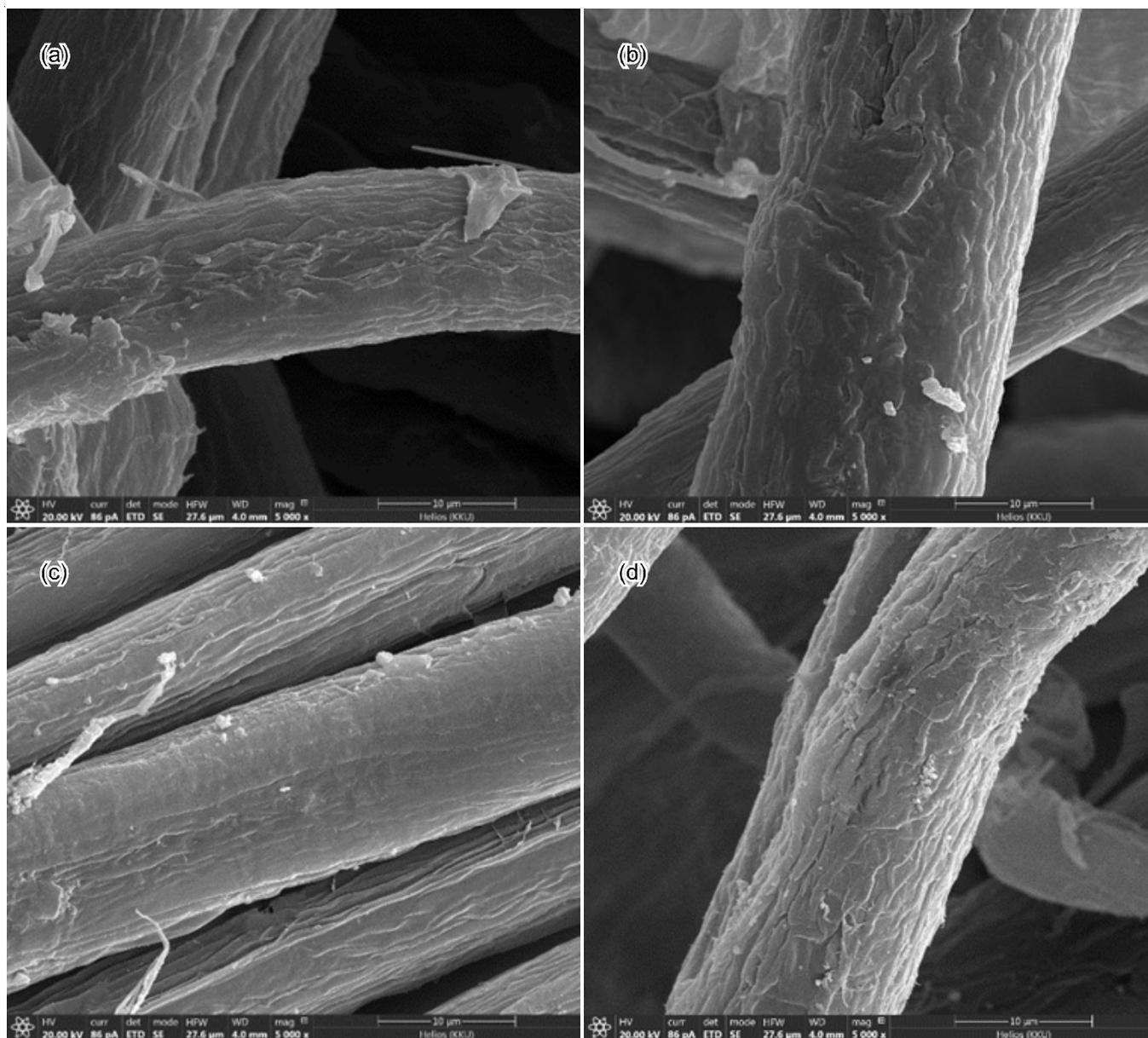


Fig. 2. SEM images of bamboo chopstick fibers (at 5000 X) (a) Cel (before adsorption), (b) Cel (after adsorption), (c) Cel-ZnO (before adsorption) and (d) Cel-ZnO (after adsorption)

**Thermal studies:** TGA was used to determine the weight loss and weight change with temperature of Cel and Cel-ZnO. It was found that Cel and Cel-ZnO began to lose its weight at 260 °C upon heating. For Cel and Cel-ZnO, the maximum weight loss occurred at 320.70 and 289.55 °C, respectively. The ash residue of Cel-ZnO after combustion was about 21.39%, which was 16.07% more than cellulose (Fig. 3). It has been found that as a result of zinc oxide embedded in cellulose, the ash content of burned wood containing Cel-ZnO is higher than that of cellulose (Table-1) [19].

#### Optimization of adsorption conditions

**Effect of pH:** The adsorption capacity of Cel and Cel-ZnO towards Cr(VI) metal ion in the  $K_2Cr_2O_7$  solution was studied at different pH values. At pH 3, both Cel and Cel-ZnO reached their maximal adsorption capacities of 122.26 and

TABLE-1  
COMPARES THE WEIGHT LOSS OF Cel AND Cel-ZnO  
UNDER CONSTANT RATE HEATING AT 10 °C/min

Sample	$T_{onset1}$ (°C)	$T_{max1}$ (°C)	$(da/dt)_{max1}$ (%/min)	Char yield at 600 °C
Cel	320.70	360.72	14.48	16.07
Cel-ZnO	289.55	340.81	10.98	21.39

253.66 mg/g when exposed to Cr(VI) solution. Since the surface of the adsorbent is protonated in a very acidic solution, the adsorption capabilities decreased with increasing solution pH (Fig. 4). However, when the surface pH is low, the adsorption capacity is also reduced due to competition between Cr(VI) ions and  $H^+$  ions. Adsorption decreases above a pH of 3, because chromium is poorly soluble in alkaline solutions and hence precipitates out [20].

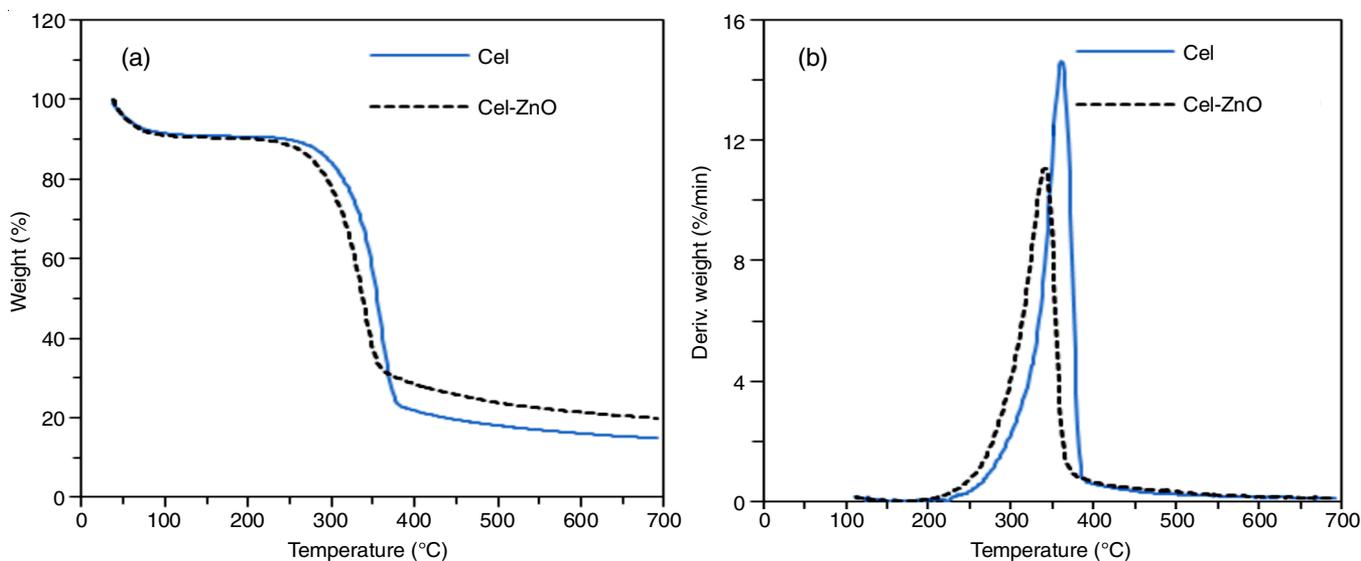


Fig. 3. The weight loss (TGA) curve (a) and weight loss with respect to derivative temperature change (DTG) (b) of Cel and Cel-ZnO sorbents

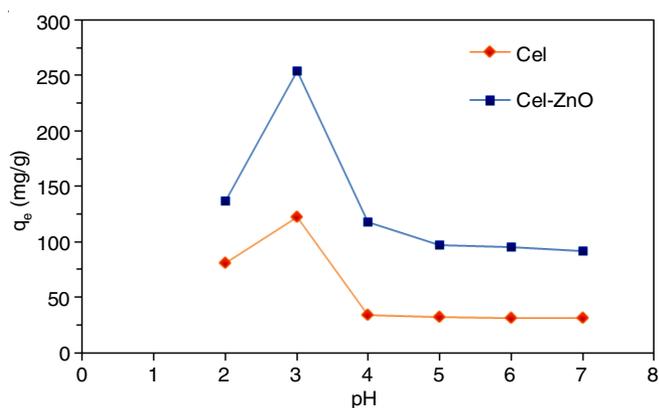


Fig. 4. Effect of pH towards the adsorption of Cr(VI) in solution

**Effect of initial concentration:** Different concentrations of dichromate solution were used to examine the adsorption capacities of Cel and Cel-ZnO sorbents towards Cr(VI) metal ion. Adsorption of 0.005 g of sorbent took 360 min at initial solution concentrations of 10, 25, 50, 100, 150, 200, 250, 300, and 500 mg/L. As can be seen from Fig. 5, both Cel and Cel-ZnO showed increasing adsorption capacities when Cr(VI) ion concentrations increases, while the latter maintained a steady adsorption capacity at 250 mg/L. Under these circumstances, the Cr(VI) adsorption capabilities of Cel and Cel-ZnO were 121.64 and 253.59 mg/g. Since, the surface area of Cel and Cel-ZnO does not change with the concentration of chromium ions in the solution, further adsorption is restricted [21].

**Effect of time and temperature:** Studies were conducted on the adsorption capacity of Cel and Cel-ZnO sorbents for Cr(VI) in a dichromate solution for 1, 3, 5, 10, 20, 30, 60, 180, and 360 min (Fig. 6). The solution concentration was set at 250 mg/L, and the adsorption temperatures were maintained at 30, 40 and 50 °C. For all of the considered reaction conditions, the adsorption efficiency of Cel did not change with the temperature. As temperatures increased, however, Cel-ZnO lost some of its adsorption efficiency. Adsorption efficiency towards Cr(VI) was shown to rapidly increase within the first 30 min,

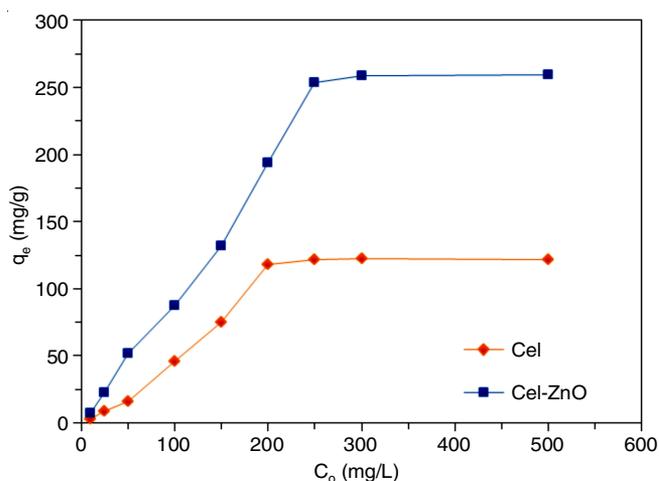


Fig. 5. Effect of initial concentration of  $K_2Cr_2O_7$  towards the adsorption of Cr(VI) in solution

before stabilizing at a high level. There were consistent patterns across all the studied three temperature ranges. Due to the high surface area of the adsorbent, the adsorption volume continues to increase until it reaches a steady state after the adsorption area volume is exhausted [22].

**Kinetics studies:** Fig. 7 displays the adsorption kinetics using pseudo-first-order and pseudo-second-order equations. By modifying the time required to attain the adsorption equilibrium while altering the reactant concentration, the adsorption mechanism could be identified. Thus, the reaction order and the correlation coefficient were calculated (Table-2). Consistent with the pseudo-second-order equation, it has been found that Cel and Cel-ZnO establish new chemical interaction during adsorption [23]. It results from the heavy metal ion and the adsorbent exchanging or giving each other electrons. Adsorption process based on Cel and Cel-ZnO sorbents responds at a rate proportional to the square of the reactant concentration.

**Absorption isotherm studies:** The adsorption isotherm was determined to correlate the amount of Cr(VI) adsorbed per weight of Cel-ZnO with the Cr(VI) concentration. When

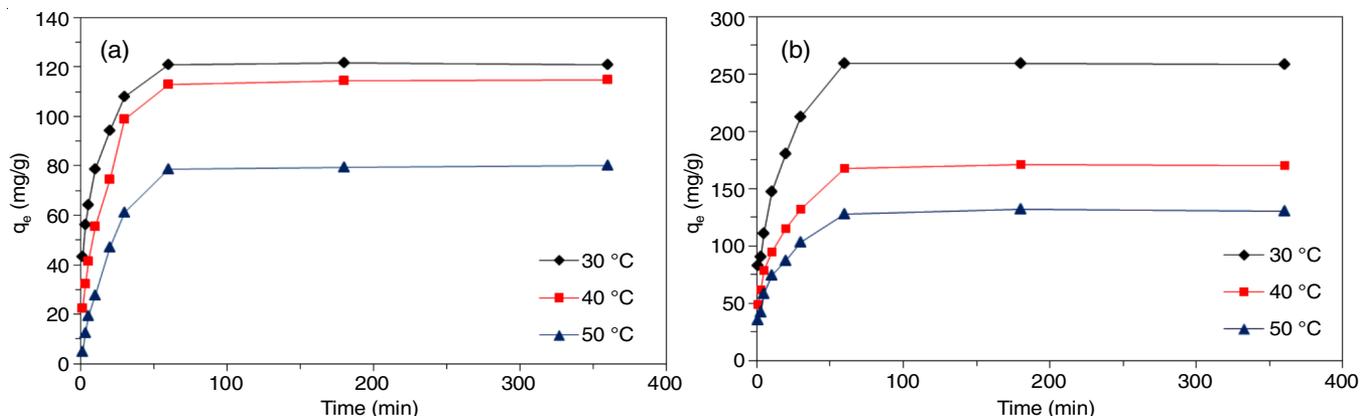


Fig. 6. Effect of time at different temperatures towards the adsorption of Cr(VI) in solution by Cel (a) and Cel-ZnO (b)

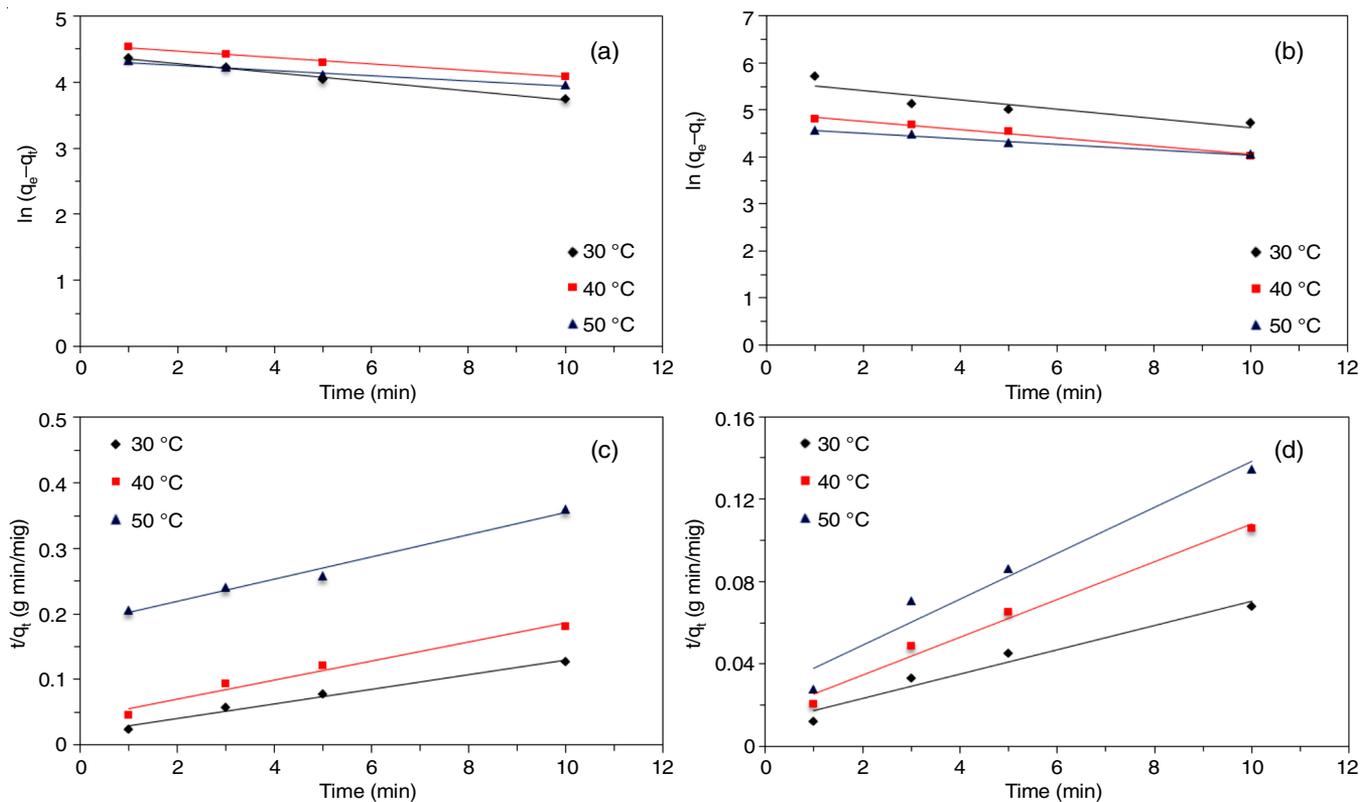


Fig. 7. Kinetics plots of pseudo-first-order and pseudo-second-order toward Cr(VI) by Cel (a-b) and Cel-ZnO (c-d)

TABLE-2  
PARAMETERS OF PSEUDO-FIRST ORDER AND PSEUDO-SECOND ORDER REACTION EQUATIONS

Sample	Adsorption temperature (°C)	$C_e$ (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo-first-order model			Pseudo-second-order model		
				$k_1$ (min <sup>-1</sup> )	$q_{e,calc}$ (mg/g)	$R^2$	$k_2$ (g/mg min)	$q_{e,calc}$ (mg/g)	$R^2$
Cel	30	250	121.2070	0.0670	82.5992	0.9930	$6.7 \times 10^3$	90.9091	0.9850
	40	250	114.8659	0.0480	95.8707	0.9910	$4.8 \times 10^3$	71.4286	0.9720
	50	250	80.2389	0.0390	76.6309	0.9720	$1.6 \times 10^3$	58.8235	0.9820
Cel-ZnO	30	250	259.1371	0.0980	271.5103	0.8120	$2.3 \times 10^3$	200.0000	0.9580
	40	250	171.0455	0.0880	139.3516	0.9830	$5.1 \times 10^3$	111.1111	0.9830
	50	250	131.7308	0.0580	102.6166	0.9820	$4.6 \times 10^3$	90.9091	0.9590

comparing the Langmuir and Freundlich isotherm graphs (Fig. 8a-d), it was found that the correlation coefficient is very near to one (Table-3). As predicted by the Langmuir model, in case of cellulose, the adsorption process is homogenous and mono-

layer in nature, with a correlation coefficient of 0.9849. The adsorption mechanism of Cr(VI) on Cel-ZnO, however, was shown to be compatible with the Freundlich model and exhibited the heterogeneous and multi-layered adsorption [24].

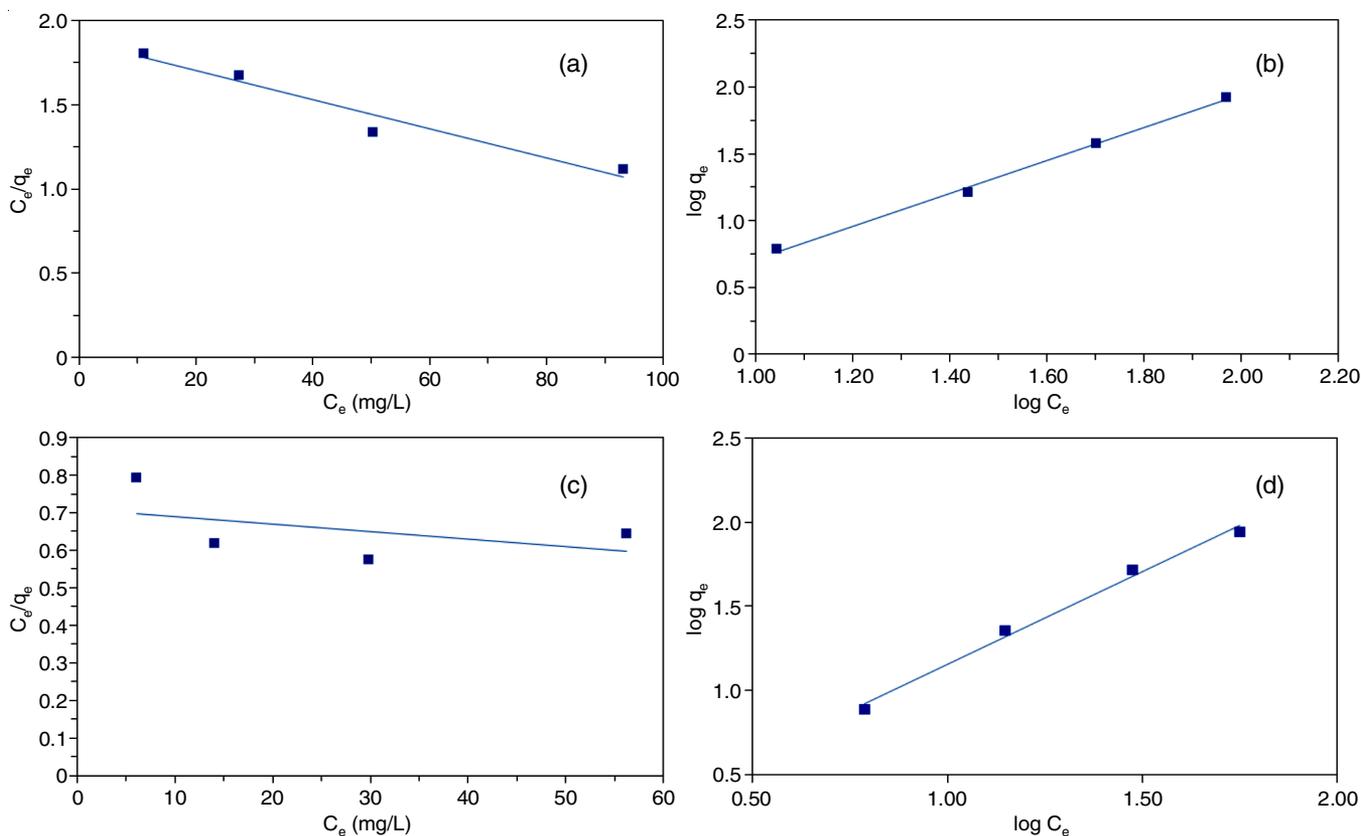


Fig. 8. Adsorption isotherm plots of Langmuir and Freundlich isotherms toward Cr(VI) by Cel (a-b) and Cel-ZnO (c-d)

TABLE-3  
PARAMETERS OF THE LANGMUIR ISOTHERM AND FREUNDLICH ISOTHERM MODELS

Adsorbents	Langmuir isotherm			Freundlich isotherm		
	$Q_L$ (mg/g)	$b$ (mL/mg)	$R^2$	$K_f$ (L/g)	$n$ (L/mg)	$R^2$
Cel	105.2632	$4.9 \times 10^{-3}$	0.9996	5.9548	32.8947	0.9308
Cel-ZnO	126.5823	$9.6 \times 10^{-3}$	0.9913	9.2618	22.6757	0.8498

**Reuseability studies:** The efficiency of Cel and Cel-ZnO adsorbents was studied. The dichromate solution was re-adsorbed with an adsorption time of 360 min, 250 mg/L initial concentration of the solution and an adsorbent amount of 0.005 g at 30 °C (Fig. 9). The Cel and Cel-ZnO adsorbents exhibited maximum efficiency for the first adsorption cycle and adsorbed less for further cycles. The adsorbents were reused for five

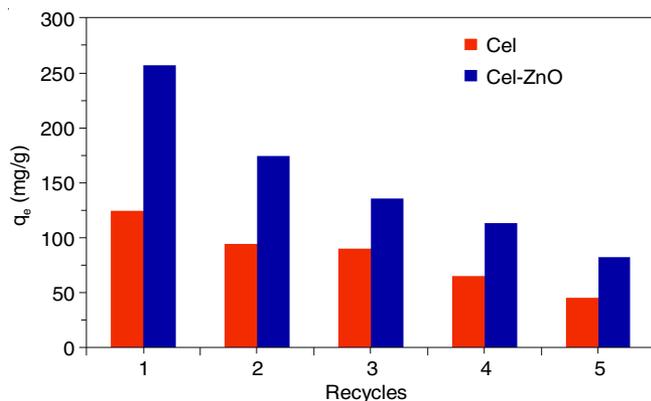


Fig. 9. Reuseability patterns of adsorption towards Cr(VI) in solutions by Cel and Cel-ZnO

consecutive cycles for the removal of Cr(VI) ions, showing that Cel-ZnO has good adsorption efficiency when reused [25].

**Comparison studies:** Table-4 provides a comparison of the maximum monolayer adsorption capacity ( $q_{max}$ ) of different adsorbents towards Cr(VI) ions with the current adsorbents. The results showed that the maximum adsorption capacity of Cel-ZnO in the present work is more than all the other ZnO obtained from different sources [26-31]. It is revealed that Cel-ZnO could be considered as a better candidate for removing Cr(VI) ions from wastewaters.

TABLE-4  
COMPARISON OF THE MAXIMUM ADSORPTION CAPACITIES OF Cr(VI) IONS ON OTHER ADSORBENT ON PREPARED FROM DIFFERENT SOURCES AT 25 °C

Adsorbent types	$q_{max}$ (mg/g)	Ref.
Cellulose-TiO <sub>2</sub> /REC	69.81	[26]
Biochar/nano-iron oxide composite	55.00	[27]
FeS/chitosan/biochar composite	103.93	[28]
Amino-functionalized magnetic cellulose	171.50	[29]
Magnetic pine cone composite	15.24	[30]
Polyvinylimidazole modified cellulose	134.00	[31]
Cel-ZnO	197.26	Present work

## Conclusion

The adsorption efficiency of the new Cel-ZnO adsorbent prepared from the used bamboo chopsticks were confirmed for the functional groups and morphological characteristics. Moreover, the optimum adsorption conditions were determined by studying the effect of various factors. By modifying the surface morphology of Cel-ZnO sorbent, base treatment renders the cellulose surface more amenable to adsorption. As a result, the surface has a tendency to uptake in Cr(VI) more effectively. It was determined that an adsorbent quantity of 0.005 g, an initial concentration of chromium of 250 mg/L, a pH of 3 and an adsorption duration of 360 min at 30 °C were optimal conditions for Cr(VI) metal ion adsorption. Compared to Cel-ZnO, which had an adsorption capacity of 91.18 mg/g, whereas the adsorption capacity of cellulose was 197.26 mg/g. So, it can be concluded that adsorption of Cr(VI) using Cel and Cel-ZnO as adsorbents showed high adsorption efficiency and offered the benefit of reusability.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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