



## Synthesize Cellulosic Nano-Composite Structure from Trash Papers and its use for heavy metal removal from Aqueous Media



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### ABSTRACT

**Background:** In this research, the adsorption efficiency of Cellulose (as a raw compound prepared from trash papers) and Cellulose Nanopolystyrene (CNP) as adsorbents for waste water treatment was investigated. Lead and cadmium ions as the pollutants added to aqueous solutions and a synthetic effluent solution was prepared.

**Methods:** The effective factors influencing the adsorption process including the pH of aqueous phase and its ionic strength, the adsorbent dose, the duration of adsorbent, and time dependency have been assessed. The removal efficiency was assessed by determination of studied metal ions using a flame atomic absorption spectrometer before and after removal procedure.

**Results:** It was found that 0.1g of Cellulose as adsorbent removed 96.81%, 50.06% of Pb<sup>2+</sup> and Cd<sup>2+</sup> from 45 mL of synthetic effluent solution at pH =5 after 30 min at 27°C, respectively. Moreover, 0.1g of the CNP removed 96.88%, 60.41% of Pb<sup>2+</sup> and Cd<sup>2+</sup> from similar above-mentioned conditions. The CNP showed better adsorbent characteristics in the adsorption of Pb<sup>2+</sup> and Cd<sup>2+</sup> ions than the Cellulose. The Langmuir and Freundlich isotherm models successfully demonstrated the experimental observations for removing Pb<sup>2+</sup> and Cd<sup>2+</sup> ions by CNP as the adsorbent. The Dubinin-Radushkevich (D-R) and Temkin isotherm models were better adapted than other used models for the adsorption procedure.

**Conclusion:** This research confirms the efficiency of treated Cellulose and Cellulose Nano-polystyrene (CNP) adsorbents from trash papers for eliminating Pb<sup>2+</sup> and Cd<sup>2+</sup> ions from synthetic effluent solution.

## 1. Introduction

During recent years, water scarcity and pollution have been one of the major problems and challenges for human societies. A variety of pollutants such as heavy metals, radioactive, organic and inorganic compounds can be mentioned as water pollutants [1- 6]. Heavy metals are typically denoted as metals or pseudo metals with a specific

gravity greater than 5g/cm<sup>3</sup>. The major hazard of heavy metals for humans is related to contact with cadmium, lead, mercury, and arsenic (arsenic is pseudo metal which is typically classified in this group) [7]. Heavy metals are known as important environmental pollutants due to their non-degradability, bioaccumulation, and the harmful effects of physiological on organisms at low concentrations. These pollutants enter the surface water resources, underground



waters, soil, biota, and the human body after production. Heavy metals in the household wastewater, industrial wastewater, and sewage sludge due to increasing the pesticides, chemical fertilizers, insecticides, and herbicides consumption in modern lifestyle are added to soil to increase agricultural production which leads to water pollution with heavy metals [1, 8]. The heavy metals are not biodegradable, thus they are found in the body fat structure of animals in the food chain. By entering them into drinking water and agriculture, they damage plant growth and cause great danger for the human and the wild life [3, 9]. Therefore, eliminating heavy metals from water ecosystems is essential work for government officials.

There are various methods for removing and separating heavy metal ions from water sources that contaminated with them. Chemical deposition, ion exchange, reverse osmosis, membrane processes, evaporation, solvent extraction, and adsorption are the most momentous methods used for this scope. Most of these techniques have defects such as sludge production or other toxic waste materials, using expensive devices, and requiring high energy and space. Among these methods, adsorption method has attracted a lot of interest in the recent years due to the simple, low cost, and executable method for eliminating heavy metal ions in different amount of contamination [10]. The adsorption technique is one of the most useful physical and chemical processes to remove contaminants and pollutants from aqueous solutions. In this way, contaminated water is passed through an adsorbent. By adsorbing it with an adsorbent, water contamination is removed. The adsorption method strongly depends on the type of used adsorbent [11,12]. Cellulose granules are a desirable adsorbent due to their properties such as hydrophobicity, high porosity, surface area, and their excellent mechanical and hydraulic properties. Paper-based products typically contain 90-99% cellulose fibers. In recent years, the chemical structure of cellulose compounds has been well recognized and consists of 2-anhydro-glucose units with predominant hydroxyl groups which are suitable functional groups for attractive reaction [13-15].

## 2. Materials and Methods

### 2.1. Chemicals and introducing of the adsorption process

#### 2.1.1. Chemicals

The initial dilutions of lead and cadmium nitrate, sodium bicarbonate, and other used salt solutions were purchased from Merck. To prepare heavy metal solutions, a stock solution of (1000 mg/L) was made using deionized water and other used solutions were obtained by appropriate dilution of this stock solution.

### 2.2. Adsorbents preparation

#### 2.2.1. Cellulose adsorbent

Newspapers waste were received from the recycling center and after crushing were washed 3 times with distilled water and boiled in a container with sodium bicarbonate to remove ink, grease, and bleach. Then the residue was washed several times with deionized water to neutralize the pH of the newspapers and the paper was dried in free air and crushed to a powdered state by a grinding machine [16, 17]. In the next step, cellulose was placed in acetone solvent for several days to separate its impurities, then the cellulose was washed with deionized water and left to dry. After drying again, it was powdered.

#### 2.2.2. Cellulose nano-adsorbents

A quantity of 0.5 grams of polystyrene (brand name One-light, used to protect electronic, electrical, and fragile containers) was dissolved in 25 mL of ethyl acetate (Merck) as organic solvent. The solution was added to a mixture of cellulose (about 1 g) and ethyl acetate (50 mL), which was previously in an ultrasonic bath for 5 min, then for producing polystyrene-stabilized cellulose, 30 mL of ethyl alcohol (96%) was sprayed using a syringe. Separating polystyrene-stabilized cellulose from the solution was done by filter paper which was washed several times with alcohol, and then washed with distilled water. The cellulose-polystyrene adsorbent became powdery after drying. The ultrasonic bath (Power sonic, Digital Ultrasonic Bath with a basket (5 liters) - UB-405) used to disperse adsorbent particles at a 350W power.

#### 2.2.3. Cellulose-nano sulfur adsorbent

After dissolving certain amount of sulfur powder in dimethyl sulfoxide (Merck) at 80 ° C, the powdered cellulose was added to and the mixture was placed in an ultrasonic bath for 15 min. By adding distilled water to the sample container, sulfur nanoparticles were formed on cellulose. Then, cellulose with nano-sized particles of sulfur (CNS) was passed through filter paper. CNS was washed twice with distilled water and straightened for removing the solvent from the adsorbent. This should be mentioned that the results of CNS adsorption showed that the S8 particles stabilization on the cellulose background reduced the adsorption efficiency; therefore, this adsorbent was put aside.

### 2.3. Adsorption process

The heavy metal solution (45 mL, initial concentrations: 20 mg/L) was placed into a 100 mL round-bottomed flask at room temperature (27°C) then 0.1g of the adsorbent was added to the heavy metal solution. The mixture of adsorbent

and heavy metal solution was shaken (300 rpm) for 30 min on a shaker (IKA KS 260). It is noteworthy that the pH of heavy metal solution was set to  $5.0 \pm 0.5$  by the addition of diluted acid or base solution ( $\text{HNO}_3$  or  $\text{NaOH}$  0.01 M). After the adsorption process, the adsorbent was stripped off by the filter paper. To determine the amount of lead and cadmium ions in the solution, the amount of the studied ions was measured before and after adsorption using a flame atomic absorption spectrometer (Varian 220 AA). For measuring heavy metals by atomic absorption, a hollow cathode lamp for lead and cadmium (with wavelengths of 212.28 and 288.8 nm respectively) was used. The number of adsorbed metal ions ( $q_e$ , mg/g) (was calculated using Eq. (1) as:

$$q_e = \frac{(C_0 - C_e)V}{M_s} \quad (1)$$

The initial and equilibrium concentration of heavy metal solutions denoted with  $C_0$  and  $C_e$  (mg/L), respectively. In addition, in this equation  $V$  (L) is the volume of the initial heavy metal solution,  $M_s$  (g) is the mass of the used fabricated or raw cellulose compounds as the adsorbent, and  $q_e$  (mg/g) is the amount of lead or cadmium ions that adsorbed on the adsorbent.

### 3. Results and Discussion

Due to importance of Functional groups in adsorption and in the structure of adsorbents (Cellulose, CNS and CNP) they were identified by FT-IR technique. The size of the particles of the used adsorbents was determined by scanning electron microscope and electron microscope image was prepared.

#### 3.1. Identification of functional groups of adsorbents

Functional groups on the surface of adsorbents are shown in Figure 1 in the form of the FT-IR spectra. Figure (1A) shows the FT-IR spectrum of cellulose which confirms the functional groups in it which are suitable for adsorbing heavy metals. The peak observed in  $3385 \text{ cm}^{-1}$  indicates OH functional group that is in the surface of adsorbents. The tape in  $2918 \text{ cm}^{-1}$  belongs to the tensile C-H and the  $1100 \text{ cm}^{-1}$  belongs to the tensile C-O group. This spectrum confirms the existence of useful functional groups that can be efficient in the process of lead and cadmium ions adsorption. In FT-IR spectrum derived from CNP (Figure. 1B), the peak observed at  $1424 \text{ cm}^{-1}$  demonstrates the presence of the  $\text{C}=\text{C}$  tensile group, which indicates the presence of polystyrene. The flattening of the peaks in the 3000 area indicates the presence of polystyrene tensile aromatic C-Hs. The FT-IR spectrum taken from the cellulose- Nano sulfur is shown in Fig. 1C. The bands appeared in the region of  $1614 \text{ cm}^{-1}$ ,  $1616 \text{ cm}^{-1}$ , and  $1668 \text{ cm}^{-1}$ , represents the presence of sulfur particles. Further, the appearance of strips in the area of  $614 \text{ cm}^{-1}$ ,  $616 \text{ cm}^{-1}$ , and  $668 \text{ cm}^{-1}$  indicates the presence of sulfur particles.

#### 3.2. SEM images

The SEM image (Figure. 2 A) shows the cellulose structure. The SEM cellulose image has long fibers visible in a Nano dimension. These strands have a width of 1 to 2 micrometers and a thickness below 100 nanometers. The existence of a Nano-sized structure in cellulose and a suitable functional group in its structure can increase the ion adsorption efficiency of heavy metals. SEM image (Figure. 2 B) refers to cellulose particles-polystyrene nanoparticles. Long fibers of cellulose fibers can be seen as a field (matrix) in Nano-composites. Spheroidal beads are polystyrene particles that appear on the cellulose field as reinforcement that is located. Beads from 100 to 900 nm can be identified in the images. The SEM image (Figure. 2 C) shows the cellulose-Nano sulfur structure in different magnifications. Sulfur particles are visible on fibrous fibers of cellulose and show Nano-particles deposited on cellulose strands.

#### 3.3. pH effect

Figure 3 shows that the metal ions removal increased as contact pH increased. This affects the adsorption amount and confirms that the adsorption depends on the pH of the aqueous solution. The data confirm that ion exchange mechanism controlled the procedure. Increasing the pH amount enhanced the negative site on adsorbents for attracting positive ions.

#### 3.4. Adsorbent dose effect

The removal of the metal ions was determined as a function of quantity used adsorbent (0.1-0.6 g). The solution pH was set to 5 and the initial concentrations of the metal ions used were 20 mg/L. Obtained data confirmed that increasing the dose of Cellulose and CNP enhanced adsorption efficiency (Figure. 4). Increasing adsorbent mass increases the active sites for adsorption and the adsorption efficiency. The increasing removal efficiency for  $\text{Cd}^{2+}$  from 84% (Cellulose), to 94% (CNP) was important point in this experiment.

#### 3.5. Time dependency and ionic strength effect

The results confirmed that heavy metal ions removal enhanced when contact time increased. The maximum removal of the heavy metal ions was obtained after 30 min of shaking time. The results showed that the relatively fast adsorption occurred during the first 30min. The solution matrix and its dissolved ions can change the efficiency of the adsorption process. This effect on the adsorption of Pb and Cd ions on the surface of Cellulose and CNP was investigated. For this purpose their removal from an aqueous phase containing NaCl (0.1-0.5 M) was studied (Fig. 5). Due to competition between the cations of the salts and lead or cadmium ions, removal efficiency was decreased.

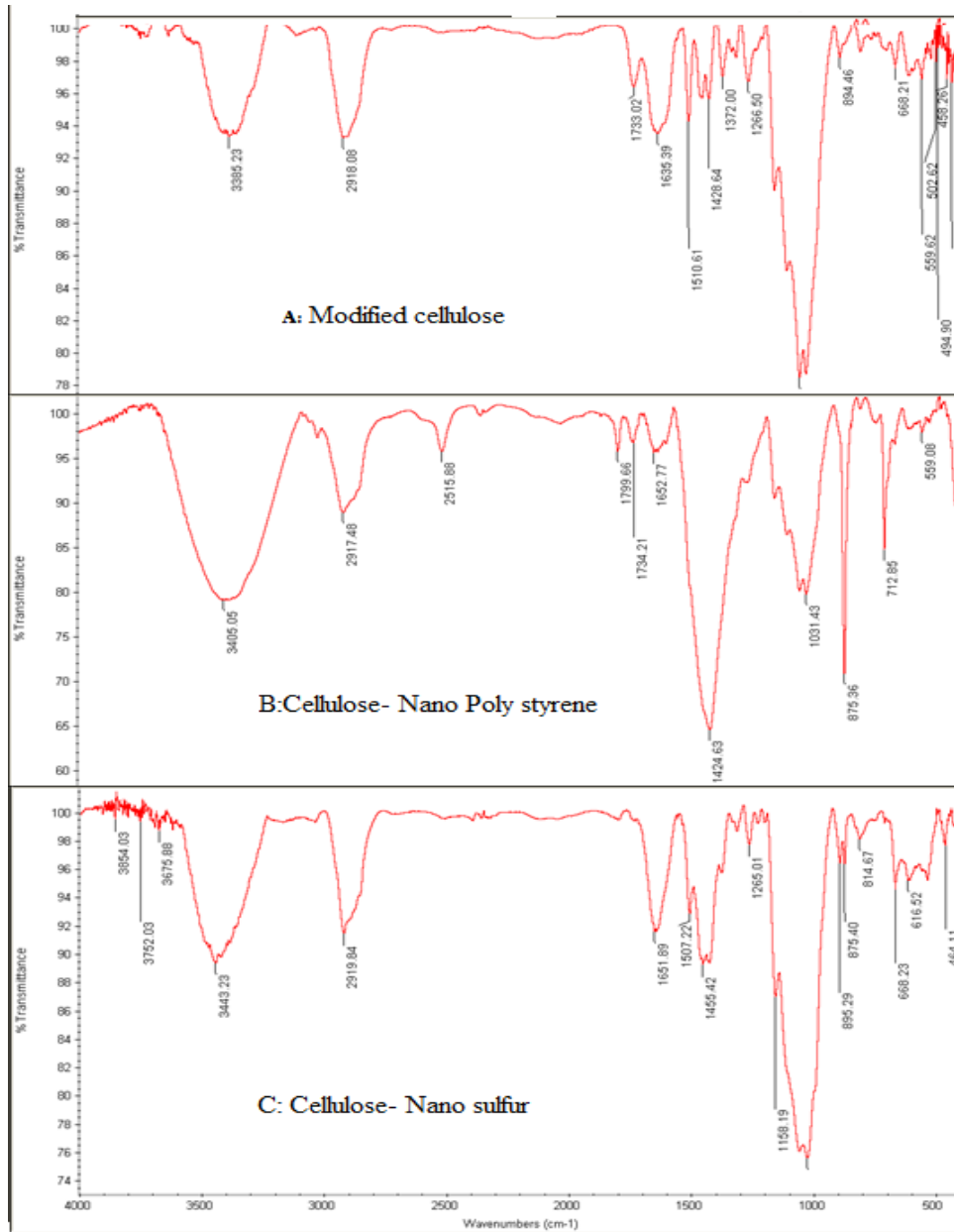


Figure 1: FT-IR spectrum of Modified Cellulose (A), Cellulose-Nano Poly styrene (B), Cellulose- Nano sulfur (C)

The second reason for this phenomenon is variation in the activity coefficients when salt concentration and ionic strength were increased. The adsorption mechanism in foreign spheres is verifiable.

3.6. Adsorption kinetic

Adsorption kinetic of the process was studied. The three useful models simple Elovich (Eq. 2), pseudo first-order (Eq.3), and pseudo-second-order (Eq. 4) kinetics were used

to survey the experimental data and examine the controlling mechanism of adsorption [18, 19]. The simple Elovich model was used for the kinetics study of the chemisorption process.

$$q_t = a + 2.303b \log t \tag{2}$$

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{3}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

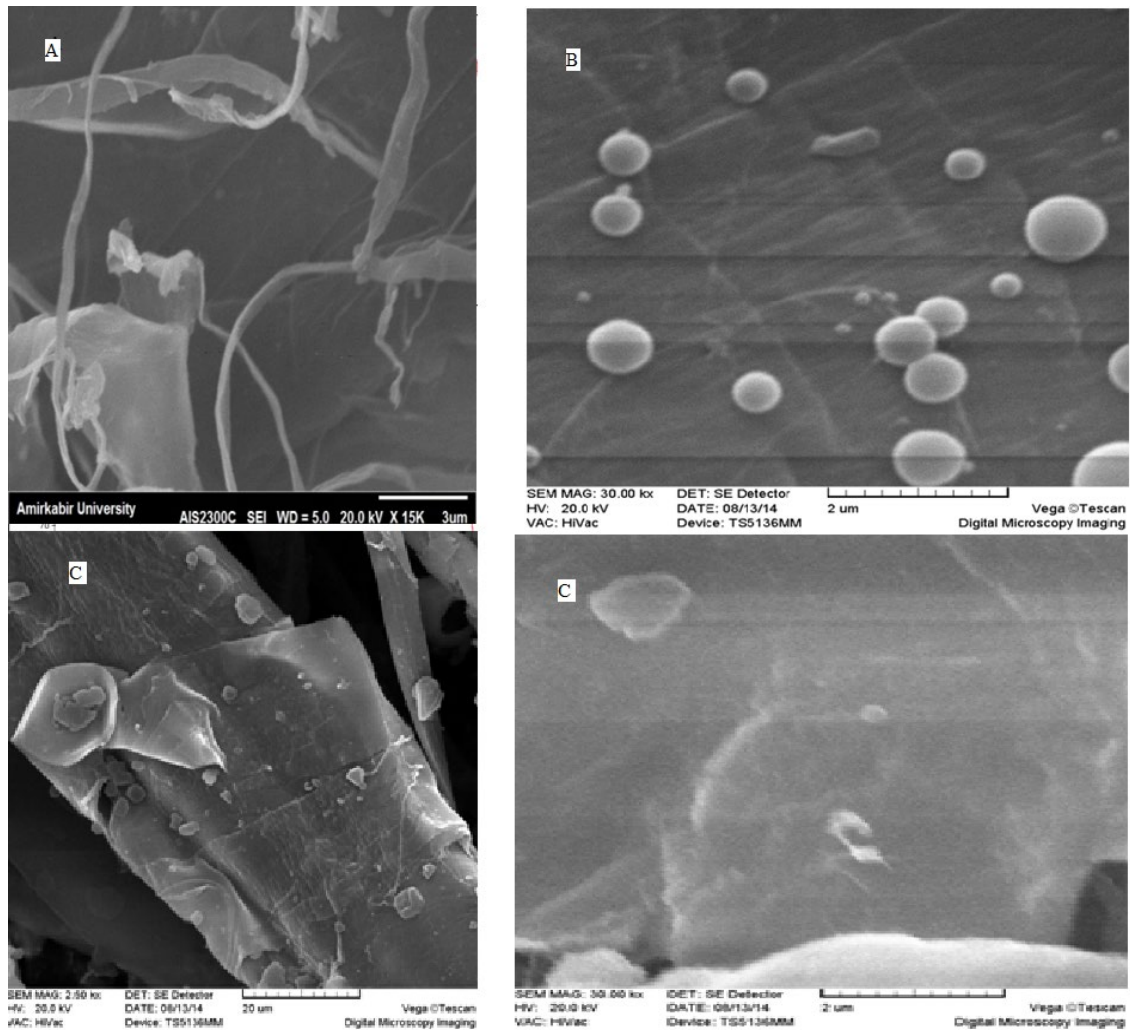


Figure 2: SEM image of modified Cellulose (A), Cellulose Nano Poly styrene (B), Cellulose- Nano sulfur (C)

Where  $a$  (mg/g. min) and  $b$  (g/mg) are both constant and represent the rate of chemisorption at zero coverage and the desorption constant for the extent of surface coverage and activation energy for adsorption, respectively.  $q_e$  and  $q_t$  (mg/g) are the amount of heavy metal adsorbed onto the surface of the adsorbent at equilibrium and  $t$  time of the adsorption process.  $k_1$  (1/min) and  $k_2$  (g/mg. min) are the pseudo-first-order and the pseudo-second-order rate constant, respectively.

The kinetic parameters for the adsorption process are given in Table 1. The pseudo-second-order model enables a significant and comparable correlation for the removal of lead and cadmium ions with cellulose and CNP adsorbents compared to the other two models.

### 3.7. Adsorption isotherms

Adsorption isotherms describe how adsorbents interact with adsorbents surface. Designing adsorption systems for

technological or practical use, describing adsorption capacity, examining the feasibility of an appropriate adsorbent and determining preliminary of the optimum amount of adsorbent is other their applications.

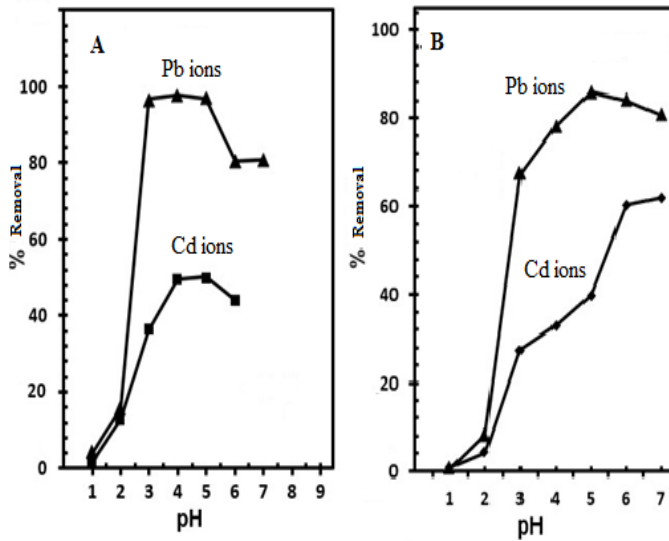
The Langmuir (Eq. 5), Freundlich (Eq. 7), Temkin (Eq. 9), and Dubinin-Radushkevich (Eq. 10) models were used to survey the adsorption isotherms [5,9,16].

$$q_e = \frac{bQ_{\max}C_e}{1 + bC_e} \quad (5)$$

Linear form of Langmuir equation is:

$$\frac{C_e}{q_e} = \frac{1}{Q_{\max}b} + \frac{C_e}{Q_{\max}} \quad (6)$$

$$q_e = KC_e^n \quad (7)$$



**Figure 3:** pH effect on the adsorption of lead (▲) and cadmium (■) ions by Cellulose (A), Cellulose- Nano Poly styrene (B)  
 Experimental conditions: Shaking rate: 300 rpm, Absorber of amount: 0.1 g, Shaking time: 30 min, Initial ion concentration: 20 mg/L, volume of solution: 45mL, Temperature: 27 °C

Logarithmic form of Freundlich model is:

$$\log q_e = \frac{1}{n} \log C_e + \log K \quad (8)$$

$$q_e = \frac{RT}{b} \ln(aC_e) \quad (9)$$

$$q_e = q_D \exp(-B_D E_D^2) \quad (10)$$

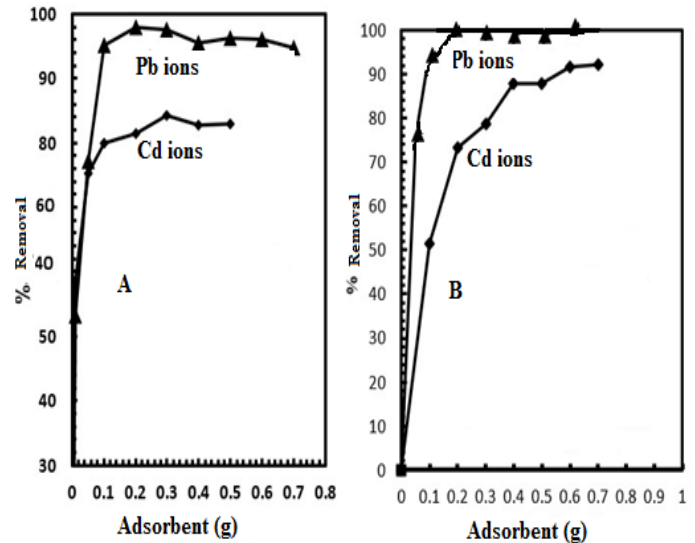
In Dubinin–Radudshkevich model (Eq. 10) ED (Polanyi potential) is calculated by:

$$E_D = RT \ln\left(1 + \frac{1}{C_e}\right) \quad (11)$$

The mean energy of adsorption process can be calculated as:

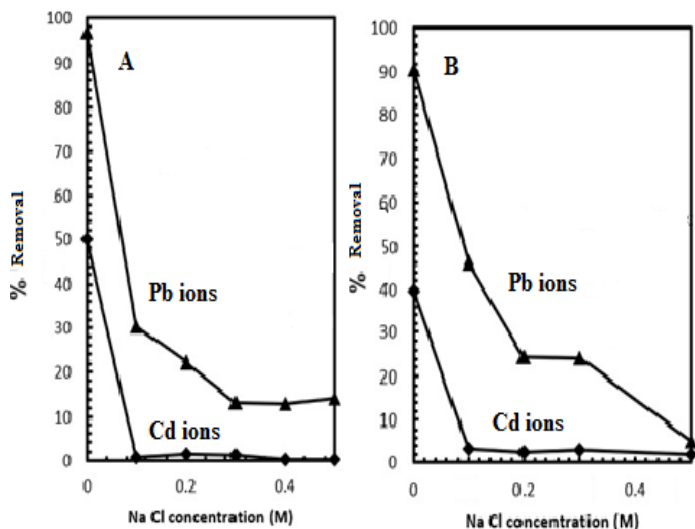
$$E = \frac{1}{\sqrt{2B_D}} \quad (12)$$

Where  $C_e$  (mg/L) is the concentration of ions in solution at equilibrium time;  $q_e$  and  $Q_{max}$  (mg/g) are the amount of ions adsorbed by 1 g of adsorbent at equilibrium and the maximum adsorption capacity of the adsorbent; and  $b$  is the constant for the binding energy of the adsorption system [16].



**Figure 4:** Effect of adsorbent amount on adsorption lead (▲) and cadmium (■) ions by Cellulose (A), Cellulose- Nano Poly styrene (B)  
 Experimental conditions: Shaking speed: 300 rpm, mixing time: 30 min, initial concentration: 20 mg/L, solution volume: 45 mL, pH=5, temperature: 27°C

$K$  and  $n$  are Freundlich constants which are relative indicators of adsorption capacity and adsorption intensity, respectively. A favorable adsorption condition is achieved when  $n > 1$  [17].  $b$  (kJ/mol) is the Temkin constant for the heat of adsorption;  $R$  constant ( $0.0083 \text{ kJ K}^{-1} \text{ mol}^{-1}$ ) denotes the gas;  $a$  (1/g) is the Temkin isotherm constant; and  $T$  (K) is the absolute temperature [9,16,17].



**Figure 5:** The effect of increasing salt (NaCl) on the adsorption lead (▲) and cadmium (■) ions by Cellulose (A), Cellulose- Nano Poly styrene (B)  
 Experimental conditions: Shaking speed 300 rpm, initial concentration (20 mg/L), solution volume: 45 mL, pH=3, temperature: 27°C, adsorbent of amount: 0.1 g

**Table 1:** Correlation coefficients and constants of kinetic equations for adsorption procedure of lead and cadmium ions on adsorbents

Cellulose- Nano Poly styrene			Cellulose			Kinetic Model
Cd <sup>2+</sup>	Pb <sup>2+</sup>	Constants	Cd <sup>2+</sup>	Pb <sup>2+</sup>	Constants	
8.065	8.772	q <sub>e</sub>	8.060	8.872	q <sub>e</sub>	Pseudo-second-order
0.067	0.271	K <sub>2</sub>	0.077	0.281	K <sub>2</sub>	
4.386	20.833	h <sub>0</sub>	4.486	19.843	h <sub>0</sub>	
0.999	1.000	R <sup>2</sup>	0.998	0.998	R <sup>2</sup>	
0.284	0.268	V	0.301	0.275	V	The power function
2.484	3.020	K <sub>p</sub>	2.380	2.996	K <sub>p</sub>	
0.612	0.488	R <sup>2</sup>	0.864	0.834	R <sup>2</sup>	
1.245	1.235	B	0.768	1.037	B	Elovich
2.657	3.755	A	2.420	3.203	A	
0.777	0.633	R <sup>2</sup>	0.612	0.704	R <sup>2</sup>	
0.046	0.035	K <sub>1</sub>	0.028	0.018	K <sub>1</sub>	Pseudo-first-order
0.926	0.444	R <sup>2</sup>	0.266	0.549	R <sup>2</sup>	

q<sub>D</sub> is the adsorption capacity of the adsorbent (mg g<sup>-1</sup>); BD (mol<sup>2</sup> KJ<sup>-2</sup>) denotes a constant for the absorption energy. The magnitude of E suggests a mechanism for the adsorption process. In fact, when E is 8-16 KJ mol<sup>-1</sup>, an ion-exchange mechanism can be considered. The dominant mechanism is physisorption for the chemisorption and ion-exchange mechanisms when E is less than 8 KJ mol<sup>-1</sup>.

The correlation coefficients and introduced adsorption isotherm constants are given in Table 2 and 3. The adsorption data for Pb<sup>2+</sup> and Cd<sup>2+</sup> ions with cellulose as a adsorbent well-fitted by the Dubinin and Temkin isotherms, respectively. Moreover, the data for Pb<sup>2+</sup> and Cd<sup>2+</sup> ions with CNP adsorbent were well - described by the Langmuir and Freundlich isotherms, respectively [2, 7, 14].

**Table 2:** Adsorption isotherm parameters for Langmuir, Freundlich, Temkin and Dubinin- isotherms (the adsorption of ions by cellulose)

Constants (Cd)	R <sup>2</sup>	Constants (Pb)	R <sup>2</sup>	Isotherms
q <sub>max</sub> (mg/g) = 87.15	0.3189	q <sub>max</sub> (mg/g) = 128.21	0.0217	Langmuir
b (L/mg) = 0.054		b (L/mg) = 0.025		
R <sub>L</sub> = 2.125		R <sub>L</sub> = 2.03		
K <sub>f</sub> (mg/g) = 58.3	0.8914	K <sub>f</sub> (mg/g) = 4.19	0.7042	Freundlich
n = 0.696		n = 1.087		
a (L/g) = 34.2	0.9724	a (L/g) = 1.37	0.7571	Temkin
b (Kj/mol) = 0.59		b (Kj/mol) = 0.2		
q <sub>D</sub> (mg/g) = 94.1	0.9197	q <sub>D</sub> (mg/g) = 1.64	0.7787	Dubinin-Radudshkevich
B <sub>D</sub> (Kj <sup>2</sup> /mol <sup>2</sup> ) = -3.75		B <sub>D</sub> (Kj <sup>2</sup> /mol <sup>2</sup> ) = 0.920		
E (Kj) = 0.37		E (Kj) = 2.32		

**Table 3:** Adsorption isotherm parameters for Langmuir, Freundlich, Temkin and Dubinin- isotherms (the adsorption of ions by CNP)

Constants (Cd)	R <sup>2</sup>	Constants (Pb)	R <sup>2</sup>	Isotherms
q <sub>max</sub> (mg/g) = 434.78	0.0	q <sub>max</sub> (mg/g) = 15.385	0.9084	Langmuir
b (L/mg) = 0.0023		b (L/mg) = 1.491		
R <sub>L</sub> = 0.96		R <sub>L</sub> = 0.0325		
K <sub>f</sub> (mg/g) = 7.78	0.97	K <sub>f</sub> (mg/g) = 1560.6	0.3527	Freundlich
n = 0.845		n = 5.6883		
a (L/g) = 1.13	0.86	a (L/g) = 3.7896	0.7003	Temkin
b (Kj/mol) = 0.58		b (Kj/mol) = 4.5061		
q <sub>D</sub> (mg/g) = -0.01	0.84	q <sub>D</sub> (mg/g) = 1.6758	0.112	Dubinin-Radudshkevich
B <sub>D</sub> (Kj <sup>2</sup> /mol <sup>2</sup> ) = 6.62		B <sub>D</sub> (Kj <sup>2</sup> /mol <sup>2</sup> ) = -0.0057		
E (Kj) = 1.95		E (Kj) = 9.3659		

#### 4. Conclusion

Cellulose and CNP as adsorbents were used to remove lead and cadmium ions from aqueous solution. Different variables such as pH, adsorbed amount, and shaking time for adsorption procedure were studied. It was confirmed that cellulose adsorbents potential is higher for lead removal than cadmium ion. The results of this research confirmed the potential of treated Cellulose (as a raw material) and Cellulose Nano polystyrene (CNP) adsorbents for removing  $Pb^{2+}$  and  $Cd^{2+}$  ions from contaminated waters. Optimized conditions for this procedure is pH = 5, shaking time = 30 min, and adsorbent dose = 0.3g (for 45 mL of sample solution with initial ion concentrations: 20 mg/L). Furthermore, it was found that the pseudo-second-order kinetic model well fitted the experimental adsorption data for all adsorbents. The data of the studied adsorption process showed that removal of  $Pb^{2+}$  and  $Cd^{2+}$  by Cellulose was well-fitted by both the Dubinin- Radushkevich (D-R) and Temkin isotherm models. However, for CNP adsorbent, the data adsorption of both studied ions is consistent with the Langmuir and Freundlich isotherm models.

#### Authors' Contributions

P.A., Researcher and prepared the manuscript. A.Z., all authors provided critical feedback and helped shape the research, analysis and manuscript. F.P., The thesis supervisor, scientific approval and paper editing. S.P., The thesis advisor, scientific approval and paper editing.

#### Conflicts of Interest

The authors report no actual or potential conflicts of interest.

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