

The Effectiveness of Removing Water Pollutants by NC (Platinum / Graphite) Prepared by Cold Plasma

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Abstract

This work aims to use of the liquid phase of nanocomposite (NC) particles (platinum / graphite) to purify water from its organic (phenol) and inorganic (lead, cadmium, chromium) pollutants. The liquid phase was prepared by exposure to cold plasma using a locally manufactured system. The pollutants are removed by adsorption method. The obtained results showed that the phenol concentration decreased by 97.6% during the first 10 minutes and by 85.7 % after 60 minutes. The lead, Cd and Cr levels decreased by approximately 90%, 89.7% and 100%, respectively, during the first 10 minutes, and by 82 %, 87% and 78%, respectively, after a period of 60 minutes. The results also exhibited that the purification percentages decrease slightly with increasing temperatures. The purification percentage of organic matter (phenol) reached about 95% at a temperature of 10 °C, and to 91% when the temperature rose to 50 °C. The lead, cadmium and chromium levels decreased about 90%, 89% and 100 %, respectively, at a temperature of 10 °C, and decreased to by 83%, 88% and 70%, respectively, when the temperature rose to 50 °C. This confirms the effectiveness of NC particles (platinum/graphite) prepared with cold plasma for use as treatment of organic and inorganic pollutants dissolved in their aqueous solutions.

Introduction:

The acceleration of population growth, the associated increase in the number of industries, factories and the consequent inevitable increase in wastewater and the dangerous organic and inorganic pollutants it contains leads to an increase in environmental pollution disturbing [1]. The problem is to develop a way of disposal of water and what it contains from pollutants in safe, effective and inexpensive ways. Note that most sewage networks (wastewater) in third world countries (including Iraq) flow directly into rivers without effective treatment [2]. This leads to surface water being more polluted due to human activity [3]. However, Iraq is facing great challenges of fresh water because of its arid and semi-arid lands, which necessitates us to think of effective solutions to recycle water to benefit from it in several areas.

Such as irrigation of crops or for various industrial uses [4]. Water is one of the most abundant natural resources on earth, covering about 70% of the Earth's surface, but only less than 3% of the water is available safe for human consumption [5]. Therefore, great care must be taken to ensure the safety of this water.

The most dangerous pollutants dissolved in water (wastewater) are: organic substances such as (phenol), which is considered one of its most important basic compounds [6], which has a very dangerous effect on human health such as cancer, reproductive abnormalities, and endocrine disorders, albeit in very low concentrations [7]. There is a high probability of the presence of trace elements, which represent inorganic elements dissolved in wastewater, such as chromium, cadmium, and lead, which have dangerous effects on health, such as affecting the rate of growth and intelligence in children, and cause a defect in the formation of neurons and imbalance In kidney function and osteoporosis, and these elements may be the cause of many types of cancer [8 – 10]. To remove these pollutants, there are several methods used, most notably ion exchange processes, the sedimentation process [11], photocatalysts [12], membrane filtration [13] and adsorption, and when comparing adsorption processes with other methods, adsorption has advantages, perhaps the most prominent of which are ease of implementation and operation, high efficiency and low cost [14].

The use of nanomaterials (NM) as an adsorbent is a very good option because of the high efficiency of nanoparticles due to the relatively large surface area of NM compared to volume [15].

Among the best options for NM as an adsorbent are platinum nanoparticles prepared by the cold plasma method because of their characteristics, the most important of which are stability and high effectiveness against adsorbents (contaminated substances dissolved in waste water) whether organic or inorganic.

Graphite is also considered one of the good materials when used as an adsorbent for pollutants due to its porous properties and other features such as its availability easily and cheaply [16]. There are materials that can be synthesized by mixing them in certain proportions in the form of a composite, where it is possible to obtain a new material with better characteristics without tampering with its basic properties for each of the materials involved in the synthesis of the composite [17].

Materials and Methods of Work

1. Prepare the Materials in the Laboratory

- 1.1. Platinum nanoparticles, ranging in size from (35-40) nanometers, were prepared by exposing aqueous platinum salts ($\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$) at a concentration of (10) mg.L^{-1} to a cold plasma system (a locally manufactured system) [18]. Where a high energy source of (6 kV) was used (in order to generate plasma) and an exposure time of (15) minutes.
- 1.2. Preparation of a NC dissolved in an aqueous solution (using deionized water). Nevertheless, it consists of graphite as a basic material at a concentration of 100

ppm and platinum nanoparticles as a reinforcing material (obtained from the previous step), a mixing ratio (1 PtNPs: 10 C) and using an ultrasonic device (It is a powerful, rack-mounted ultrasonic homogenizer designed for use in bulk or continuous processes that delivers 400 watts of reliable 24 kHz ultrasonic power to lab samples. Its uses include homogenization, dispersion, and other uses). For the mixing process with a capacity of (100) watts and a duration of mixing (30) minutes to get a superimposed, as shown in Fig. 1. (a). This NC has an zeta potential (an examination that is used to know the cell's electricity to understand some biological systems) of less than (30) mV, as shown in the fig. 1. (b).

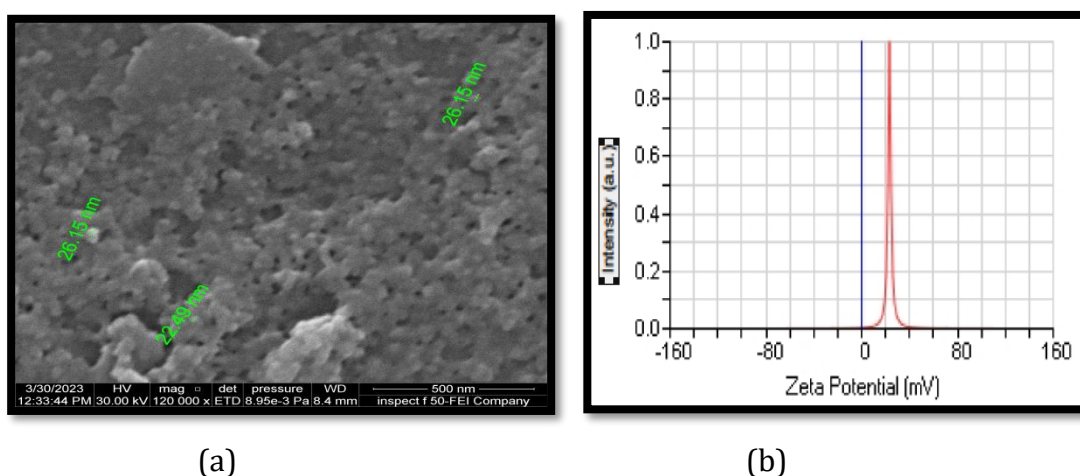


Fig. 1. (a)(Platinum/ Graphite) NC.(b) The zeta potential of a NC (platinum/graphite).

- 1.3. Prepare the organic pollutant (phenol) in the laboratory at a concentration of (100) mg. L⁻¹.

The standard solution was prepared with a concentration of (1000) mg. L⁻¹ of phenol, where phenol was added with a weight of (1) gram, and its purity (99%) to a volumetric vial of 1-liter capacity, add half a liter of deionized water to it. It was shaken well until the substance dissolved for (15) minutes, then the volume was completed with distilled water to the mark of the solution, and then the solution was diluted to (100) mg. L⁻¹.

- 1.4. 4- Preparation of inorganic pollutants (lead, cadmium, chromium) in the laboratory at a concentration of (10) mg. L⁻¹. Using the equation below to find the weight of lead, cadmium, chromium required to obtain it at a concentration of (1000) mg L⁻¹ dissolved in its aqueous solution. and then the solution was diluted to (10) mg.L⁻¹ [19].

$$W. = (Con./1000) \times (M.W.C./M.W.ELE.) \times \left(\frac{V}{1000}\right) \quad (1)$$

Where:

w.= Weight (in grams)

Con.= Concentration

m. w. c. = The molecular weight of the compound.

$m.w.ele.$ = Partial weight of the element

V = Required volume

Deionized water was used as a solvent for the above pollutants to ensure that there are no other substances that may affect the treatment and disposal of pollutants.

2. Adsorption Experiments

Treating the dissolved phenol with its aqueous solution at a concentration of (100) mg.L^{-1} , and treating heavy metals (lead, cadmium, and chromium) at a concentration of (10) mg.L^{-1} and as shown below:

Adsorption processes for organic and inorganic pollutants (phenol, lead, cadmium, and chromium) were carried out. Dissolved in its aqueous solution and to obtain adsorption isotherms for the prepared solutions, (5) ml of the colloid containing the NC (platinum/ graphite) for each sample was placed in volumetric bottles of (25) ml, and (5) ml was added to each sample of the prepared solutions (organic and inorganic pollutants) at a concentration of (100) mg.L^{-1} and a concentration of (10) mg.L^{-1} , respectively. Volumetric bottles were closed, then placed in a rocking water bath with a temperature of (25) $^{\circ}\text{C}$, and for different periods of time of continuous shaking (10- 60) minutes for a period of (10) minutes, noting that the samples were immersed in water for about half within the water bath, then the samples were taken out one by one, and the above samples were separated by Centrifuge (5000) rpm for (10) minutes.

The absorbance of the phenol solution was measured by a spectro-visible-ultraviolet spectrometer, then the equilibrium concentration was determined from the calibration curve shown in Fig. (2).

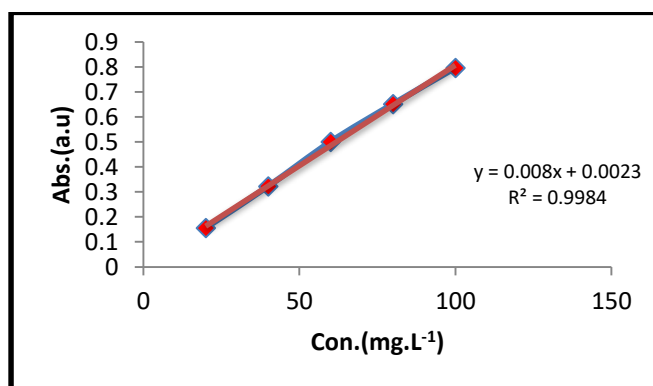


Fig. 2. Standard calibration curve for phenol solutions prepared at wavelength (270) nm

- The concentration of the adsorbent (phenol) was calculated for different time periods according to the mathematical relationship(2) below [20]:

$$con. = \frac{Abs.-Intercept}{slope} \quad (2)$$

where:

Con. = concentration

Abs. = absorbance

- The concentrations of the adsorbents (lead, cadmium, and chromium) were calculated using the atomic absorption spectrometer.
- The percentage of the pollutant concentration after treatment (pollutant removal percentage) was calculated to find out the adsorption efficiency according to the mathematical relationship (3) below"[21]".

$$\text{Removal Efficiency } R\% = \left[\frac{(C_o - C_e)}{C_o} \right] \times 100 \quad (3)$$

Where:

C_o = initial concentration (mg/L)

C_e = concentration at equilibrium (mg/L) and is determined according to the duration and time of adsorption.

3. Calculating Thermodynamic Functions

The values of the equilibrium constant (K_{eq}) of adsorption were calculated for the adsorbent (amount of removal of the pollutant) at different temperatures (10, 20, 30, 40, and 50) °C, so when the state of equilibrium occurs between each of the adsorbent and the remaining substance (i.e. the non-adsorbent) in the solution. The value of the equilibrium constant for adsorption can be found by equation (4) [22]:

$$K_{eq} = \frac{\chi_{eq}}{a - \chi_{eq}} \quad (4)$$

Where:

K_{eq} = equilibrium constant.

$a - \chi_{eq}$ = concentration of the free (non-adsorbed) substance at equilibrium.

χ_{eq} = amount of substance adsorbed at equilibrium.

a = initial concentration.

From knowing the values and quantities of the thermodynamic functions, we will know the nature and type of the adsorption process (physical or chemical), and therefore this leads us, through its computational values, to know the behavior of NC inside aqueous solution. The most important of these functions are:

- 3.1. Gibbs energy (active or free energy), A thermodynamic property that combines the entropy and enthalpy of a system in order to determine whether a reaction is spontaneous and is symbolized by (ΔG). It is calculated by relation (5) below:

The values of (ΔG) were calculated from the equation [23]:

$$\Delta G = - RT \ln K_{eq} \quad (5)$$

Where:

R = the general gas constant.

T = absolute temperature.

- 1- The enthalpy function, symbolized by (ΔH), represents the enthalpy of the system, and the value (ΔH) is calculated from the (vant-Hoff) equation"[24]":

$$K_{eq}=K_o e^{-\Delta H/RT} \quad (6)$$

$$\ln K_{eq} = \ln K_o - \Delta H/RT \quad (7)$$

By drawing the relationship between ($\ln K_{eq}$) as a function ($1/T$), a straight line with a slope equal to ($-\Delta H /R$) is obtained.

- 3.2. Entropy, symbolized by (ΔS), and its value is equal to the amount of work done on the system, meaning the largest randomness of the system, The value of (ΔS) is calculated from the following equations[25]:

$$\Delta G = \Delta H - T\Delta S \quad (8)$$

$$\Delta S = (\Delta H - \Delta G) / T \quad (9)$$

From the values of these thermodynamic functions, it is possible to know whether the reaction is absorbent or heat-emitting (chemical or physical). In other words, there are new products and compounds that were obtained during the adsorption process (if it was chemical) or there were no new products and compounds in the sense of (physical reaction), i.e. It's a safe interaction [24 - 25].

Results and Discussion:

Removal of pollutants by NC

1. remove the organic pollutant (phenol):

The effect of time on the removal of the pollutant (phenol):

The removal percentage of (phenol) dissolved in water was measured at a concentration of (100) mg.L⁻¹, at a (λ_{max}), and temperature of (25) °C, after mixing it with the solution containing the NC prepared with cold plasma to obtain an adsorption process for the pollutant (phenol). The adsorption time for the polluted material is from (10-60) minutes for a period of (10) minutes, as shown in Fig. (3).

Calculating the best adsorption efficiency percentages (%R) for the pollutant (phenol), and calculating the equilibrium concentration as shown in Table (1).

Let's find that the best processing time is in the first 10 minutes.

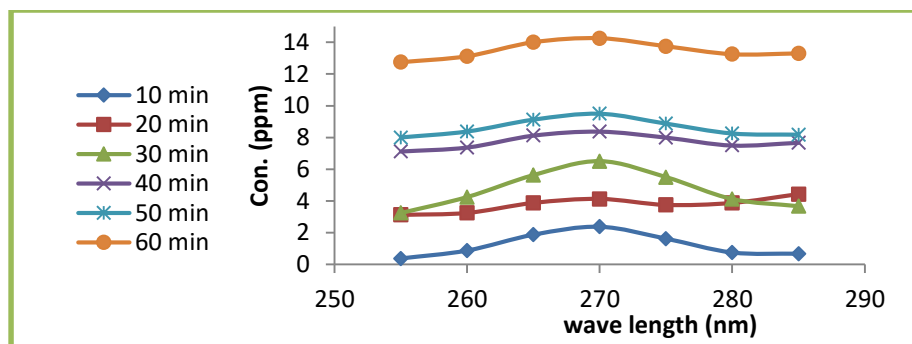


Fig. 3. Concentration of phenol after treatment with NC particles prepared by cold plasma method as a function of time

Table 1: Removal percentage and equilibrium concentration of phenol at an initial concentration of 100 mg. L⁻¹ as a function of time

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	Adsorption time (min)
97.625	2.375	10
95.875	4.125	20
93.5	6.5	30
91.625	8.375	40
90.5	9.5	50
85.75	14.25	60

• **The effect of temperature on the removal of organic pollutants (phenol)**

Platinum/graphite nanoparticles in their liquid phase prepared in cold plasma were sampled to study the effect of temperatures at (10, 20, 30, 40, 50) °C on the amount of adsorbent for 10 minutes. Fig. (4) and Table (2) show the percentage of contaminant removal at these different temperatures, with the removal percentages shown as a function of temperature. We note that at lower temperatures (10, and 20) °C, material removal was the best. But at higher temperatures, we observe lower removal rates, which means lower adsorption efficiency.

The rising of temperature increases the energy of the absorbed part, which helps to separate it from the absorption surface and return it to the inside of the solution.

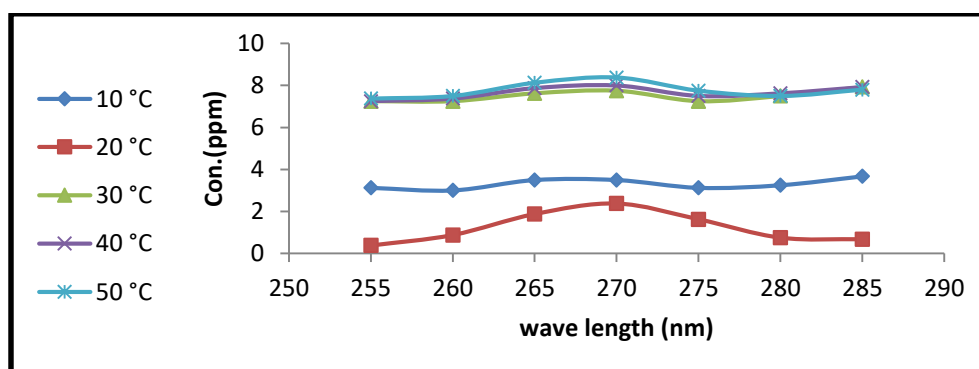


Fig. 4. Phenol concentrations after treatment with NC particles prepared by cold plasma method by temperature change.

Table 2: Removal ratio and equilibrium concentration of the contaminant (phenol) with an initial concentration of 100 mg.L⁻¹ as a function of temperature

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	Temperature at adsorption (°C)
96.5	3.5	10
97.625	2.375	20
92.25	7.75	30
92	8	40
91.625	8.375	50

2. Removal of Inorganic Pollutants (Lead, Cadmium, Chromium):

The experiment was conducted on heavy elements with different positions within the periodic table, meaning the atomic number, electronic distribution, and different chemical properties, to see the ability of the prepared material (NC) to remove these pollutants from their aqueous solutions.

• Effect of time on material removal inorganic pollutants

The removal percentage of inorganic pollutants (lead, cadmium, and chromium) dissolved in water was measured at a concentration of 10 mg.L⁻¹, at a temperature of 25 °C. After mixing each of them with a solution containing NC (platinum / graphite) in a mixing ratio (1:1) to obtain an adsorption process for the pollutant. The adsorption time for the polluted material was from (10-60) minutes for a period of 10 minutes, and the adsorption efficiency and equilibrium concentration were calculated as shown in Tables (3,4, and 5).

From the observation of the mentioned tables, pollutants with a lower atomic number (chromium) Table (5) achieved the best adsorption efficiency (100%) from the first 10 minutes due to the speed and ease of withdrawing adsorbed particles (chromium), due to the small size of chromium atoms, the number of adsorbed atoms is more relatively compared with lead and cadmium, according to Langmuir's proposal [25], note that the adsorption efficiency of lead and cadmium pollutants (3 and 4), respectively, is good, achieving rates of approximately 90%. These are encouraging ratios, and the reason for the high adsorption efficiency of all inorganic pollutants (lead, cadmium, chromium) dissolved in their aqueous solutions is that the zeta potential of the adsorbent (NC) is active and unstable[26].

Table 3: Removal percentage and equilibrium concentration of the pollutant (lead) with an initial concentration of 10 mg.L⁻¹ as a function of time.

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	Adsorption time (min)
90.040	0.996	10
89.984	1.002	20
86.493	1.351	30
84.185	1.581	40
83.672	1.633	50
82.857	1.714	60

Table 4: Removal percentage and equilibrium concentration of the pollutant (cadmium) at an initial concentration of 10 mg L⁻¹ as a function of time

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	Adsorption time (min)
89.791	1.021	10
89.094	1.091	20
88.536	1.146	30
88.443	1.156	40
88.443	1.156	50
87.746	1.225	60

Table 5: Removal percentage and equilibrium concentration of the pollutant (chromium) at an initial concentration of 10 mg L⁻¹ as a function of time

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	Adsorption time (min)
100	0.000	10
89.62	1.038	20
89.62	1.038	30
89.62	1.038	40
85.77	1.423	50
78.077	2.192	60

•The effect of temperature on the removal of inorganic pollutant

Samples were taken from the NC (platinum / graphite) in its liquid phase and were prepared with cold plasma to study the effect of temperatures at (10, 20, 30, 40, and 50) Celsius on the amount of adsorbent material and mixed in a ratio (1:1) with inorganic pollutants (lead, cadmium, and chromium) for an adsorption period of 10 minutes. This time is the best adsorption time obtained, so the removal rate of the pollutant material was better at lower temperatures, but at higher temperatures, we notice a decrease in the removal rates, as shown in Tables (6, 7, and 8).

This means that the adsorption efficiency is less effective. It was also noted that the pollutant with the smallest crystal size and the lowest atomic number is less affected by the high temperatures.

Table 6: Removal percentage and equilibrium concentration of the pollutant (lead) at an initial concentration of 10 mg.L⁻¹ as a function of temperature

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	temperature at adsorption (°C)
90.095	0.991	10
90.040	0.996	20
84.518	1.548	30
84.145	1.586	40
83.398	1.660	50

Table 7: Removal percentage and equilibrium concentration of the pollutant (cadmium) at an initial concentration of 10 mg.L⁻¹ as a function of temperature

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	temperature at adsorption (°C)
89.698	1.030	10
89.791	1.021	20
88.862	1.114	30
88.304	1.170	40
88.304	1.170	50

Table 8: Removal percentage and equilibrium concentration of the pollutant (chromium) at an initial concentration of 10 mg.L⁻¹ as a function of temperature

Removal Efficiency (R%)	Equilibrium concentration (mg/L)	temperature at adsorption (°C)
100	0.000	10
100	0.000	20
89.615	1.038	30
85.769	1.423	40
70.385	2.962	50

Calculation of Thermodynamic Functions of Adsorption

The values of the thermodynamic functions of the adsorption process that were carried out by adsorbed NC particles were calculated at the highest adsorption and best removal of the pollutant for the best time, which is 10 minutes, in a range of temperatures ranging from (10-50)°C, and a range of 10°C.

By calculating the thermodynamic functions: enthalpy (ΔH), entropy (ΔS), Gibbs free energy (ΔG), as well as calculating the Equilibrium Constant (K_{eq}), it is possible to find out whether adsorption by NCs prepared by the cold plasma method of contaminated materials Organic (phenol) and inorganic (lead, cadmium, and chromium) is a physical or chemical adsorption [27], as shown below:

- 1- Calculating the equilibrium constant (K_{eq}) and the reaction constant (k): equation no. (5,6), the high values of the reaction speed (k) indicate that the adsorption efficiency is high, as shown in Tables (9,10,11,and12). for pollutants Phenol, lead, cadmium, and chromium respectively Observing these tables, we find that the values of the reaction speed (k) decrease with increasing temperature. Because the rise in temperature works to return the adsorbed material from the adsorbent surface to the adsorption solution, and this is consistent with the physical nature of heat-emitting adsorption [28].
- 2- Gibbs energy (ΔG), or it is called the activation free energy values. When calculated from equation no. (5), the negative values show that the reaction proceeds automatically towards the formation of products during the adsorption processes, in the sense of a physical interaction, as shown in Table (9,10,11,and 12) for pollutants Phenol, lead, cadmium, and chromium respectively [22].
- 3- The enthalpy (ΔH) through adsorption processes using the (Vant- Hoff equation) equation no. (6, 7), and drawing the relationship between ($\ln K_{eq}$ as a function of

the reciprocal of temperature ($1/T$) K^{-1}). From the slope value that represents $-\Delta H / R$ as shown in Fig (5,6,7, and 8) for pollutants Phenol, lead, cadmium, and chromium respectively, the calculated value (ΔH) was negative and less than (40 KJ/mol) indicating that the reaction is exothermic (physical) during the adsorption processes as shown in Table (9,10,11, and 12) for pollutants Phenol, lead, cadmium, and chromium respectively [29].

- 4- Entropy values (ΔS) and it is noted through the values of change (ΔS) equation no. (8, 9), the random state or disorder in the adsorption system refers to the values (ΔS) in Table (9,10,11, and 12) for pollutants Phenol, lead, cadmium, and chromium respectively almost do not change (there is no significant change) because the state of disorder for one compound It does not change within the low temperature range[30].

Table 9: Thermodynamic values of (phenol) adsorption process on the surface of NC particles

ΔS	ΔH	ΔG	slope	$\ln K$	K	$T^{-1}(K)$
62.581		-21670.425		9.210	9999.000	0.00353
63.058		-22436.164	2858.957	9.210	9999.000	0.00341
4.853	-32110	-5430.508		2.156	8.634	0.00330
2.282		-4674.523		1.796	6.027	0.00319
-5.065		-2324.124		0.865	2.376	0.00310

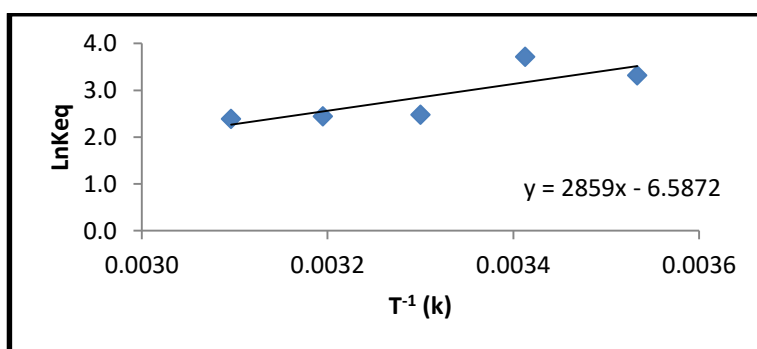


Fig. 5. The linear relationship of the Vant-Hoff equation for phenol adsorption by the NC

Table 10: Thermodynamic values of (lead) adsorption process on the surface of NC particles

ΔS	ΔH	ΔG	slope	$\ln K$	K	$T^{-1}(K)$
-80.330		-5193.390		2.207	9.091	0.00353
-77.008	-13157.1	-5363.289	1582.5291	2.202	9.040	0.00341
-78.294		-4203.647		1.669	5.305	0.00330
-75.349		-4342.381		1.669	5.305	0.00319
-73.040		-4334.931		1.614	5.024	0.00310

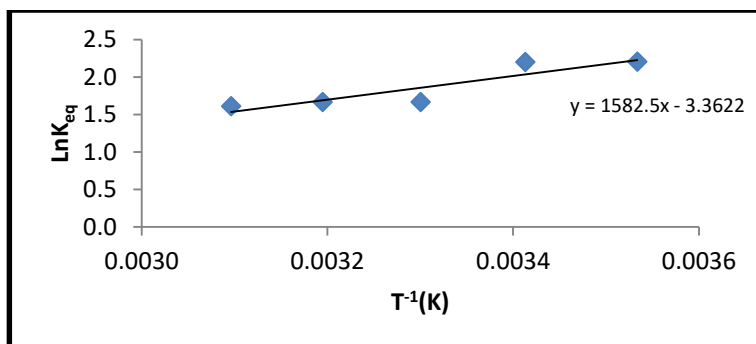


Fig. 6. The linear relationship of the Vant-Hoff equation for (lead) adsorption by the NC

Table 11: Thermodynamic values of (cadmium) adsorption process on the surface of NC particles

ΔS	ΔH	ΔG	slope	$\ln K$	K	$T^{-1}(K)$
15.459	-3339.75	-5092.362	401.702	2.164	8.709	0.00353
15.627		-5296.127		2.174	8.794	0.00341
14.896		-5231.048		2.077	7.977	0.00330
14.512		-5259.606		2.021	7.547	0.00319
14.583		-5427.644		2.021	7.547	0.00310

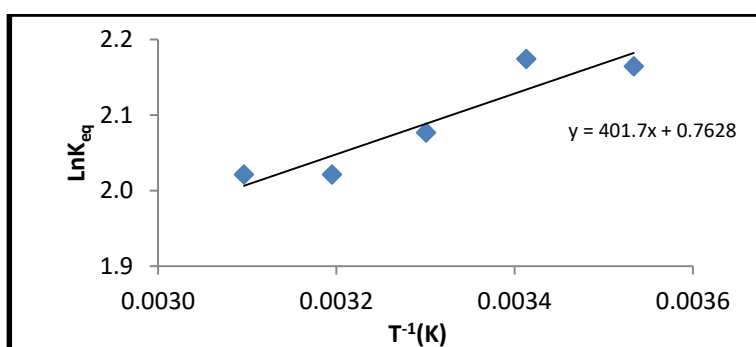


Fig. 7. represents the linear relationship of the Vant-Hoff equation for (cadmium) adsorption by the NC

Table 12: Thermodynamic values of (chromium) adsorption process on the surface of NC particles

ΔS	ΔH	ΔG	slope	$\ln K$	K	$T^{-1}(K)$
62.581	-184166	-21670.425	22151	9.210	9999.000	0.00353
63.058		-22436.164		9.210	9999.000	0.00341
4.853		-5430.508		2.156	8.634	0.00330
2.282		-4674.523		1.796	6.027	0.00319
-5.065		-2324.124		0.865	2.376	0.00310

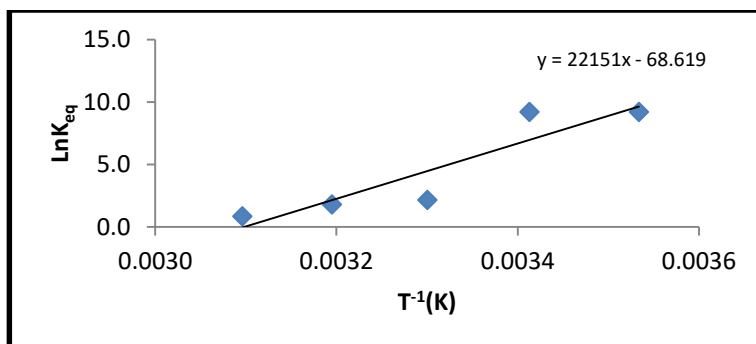


Fig. 8. The linear relationship of the Van-Hoff equation for (chromium) adsorption by the NC

CONCLUSIONS

The use of NC particles (platinum / graphite) in its liquid phase prepared by the cold plasma method was conducted to remove dissolved pollutants with its aqueous solution, whether organic (phenol) or inorganic (lead, cadmium, chromium) at a temperature of (25) °C. Pollutant purification rates were about (97%) for organic matter (phenol) and up to (90% - 100%) for inorganic substances (lead, cadmium, chromium) despite their different positions in the periodic table (they have different atomic number, electronic distribution, and chemical properties). Furthermore, the purification efficiency of these dissolved pollutants in their aqueous solutions remains at their high levels, with a very slight decrease in the purification efficiency at high temperatures for aqueous solutions containing pollutants, which indicates the effectiveness of the NC in the purification process. The purification of these pollutants was carried out in a very safe, easy and inexpensive way, because it was done by the physical adsorption method, and this was proven through the values of the thermodynamic functions obtained during the purification process. It should also be noted that the NM used for processing were also discarded during the sample separation process by the centrifuge used.

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