

**Article**

**The Study of Using Gold Nanoparticles as an Adsorbent for Water Treatment**

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**Abstract**

In this study gold nanoparticles were prepared by wet chemistry method. The source of gold is H<sub>2</sub>AuCl<sub>4</sub> and D- glucose sugar used as a reducing agent and tri sodium citrate used as masking agent. The prepared gold nanoparticles were used to remove the lead ion from the contaminated aqueous solutions. The prepared gold nanoparticles were identified through Transmission Electron Microscope (TEM). The TEM images showed that the average diameters of gold nanoparticles ranged from 50-90 nm and dynamic light scattering (DLS) represented by zeta potential and zeta sizer analyzer device used to examine the stability and size distribution of gold nanoparticles. The plasmon resonance band for gold nanoparticles was found in the wavelength 530 nm. Various conditions affecting the adsorption process were studied, such as equilibrium time, temperature, acidity function and initial concentration of lead ion. Adsorption increases with increasing temperature, meaning that the reaction is endothermic. The adsorption isotherms of Langmuir and

Freundlich were also studied. The Langmuir equation was the most applicable to the studied system. The thermodynamic studies showed that the removal process was spontaneous, and positive values of  $\Delta S$  indicate for increasing in randomness.

**Keywords: Adsorption, Gold nanoparticles, Thermodynamic studies, water treatment**

## **Introduction**

In recent years, nanotechnology field and its applications have been expanded, entering into many fields such as biology, environment, energy, and other<sup>1-3</sup>. Among the metallic nanoparticles, gold and silver nanoparticles are the most important because of their wide applications<sup>4</sup>. In general, nanomaterials can be defined as particles whose size is less than one hundred nanometers (A nanometer is one thousandth of a millionth of a meter) which is given to the material in its composition. New properties and behaviors that differ from the bulk<sup>5</sup>. Among these characteristics is the surface area, where the surface area of nano materials increases when compared with the same mass of the material in the larger scale, and this makes the nano materials more chemically active and also affects their strength<sup>6</sup>. As a result of these properties, adsorption capacity and extraordinary effectiveness, nano materials are used to remove pollutants, heavy metal ions. Extensive studies have been conducted on the application of nano materials in the treatment of heavy mineral water, and they have shown countless potential in the removal of heavy metals<sup>7</sup>. The presence of heavy metal ions in the ecosystem is of attention due to its

toxicity to life. When toxic compounds come to the surface of the earth, they do not pollute not only surface water, but it also arrives in small quantities below the surface of the water by leaching from the soil after the fall rain and snow, so surface water contains various toxic elements<sup>8-11</sup>.

Therefore, great efforts have been made by many researchers to remove ions dissolved and suspended heavy metals from industrial wastewater different types of techniques have been used to accomplish This objective, including: ion exchange, solvent extraction, chemical precipitation, reverse osmosis, and adsorption. But adsorption has become the preferred method for removing toxic heavy metals and recovering them from waste water<sup>12-14</sup>.

## **Materials and methods**

### **Preparation of gold nanoparticles**

Gold nanoparticles were prepared by mixing 1000 mg. L<sup>-1</sup> from H<sub>2</sub>AuCl<sub>4</sub> as a source of gold ions with the same volume of D-glucose sugar with the same concentration 1000 mg. L<sup>-1</sup> as a reducing agent, and then sodium citrate was added to prevent aggregate of the prepared gold nanoparticles. The solution stirred for half an hour at 65 °C until its color changes to purple, as the purple color indicates the presence of gold nanoparticles.

## **Preparation of lead stock solution**

A series of standard solutions of lead were prepared by diluting from standard solution at a concentration of 1000 mg. L<sup>-1</sup> with deionized water in the range 1 - 20 mg L<sup>-1</sup>.

## **Adsorption studies**

The lead ions adsorption on the gold nanoparticles surface. The adsorption was studied using the batch method, several factors such as contact time, pH, the initial concentration of the adsorbent and the adsorption isotherm have been studied. The study was investigated using 100 ml covered conical flasks, where (0.01g) of gold nanoparticles were added to the 15 ml of lead ion. It was shaken until equilibrium was reached at a rate of vibration per minute. The concentration of lead was estimated using a flame atomic absorption device. The removal efficiency and the equilibrium uptake of lead were found according to the Eq. 1 and Eq. 2.

$$Removal \% = \frac{C_0 - C_e}{C_0} * 100 \dots\dots\dots 1$$

$$Q_e = V_{sol} * (C_0 - C_e) / m \dots\dots\dots 2$$

Hence,

C<sub>e</sub> is the concentration of lead ion in the equilibrium (mg. L<sup>-1</sup>),

C<sub>0</sub>: Lead ion initial concentration (mg. L<sup>-1</sup>),

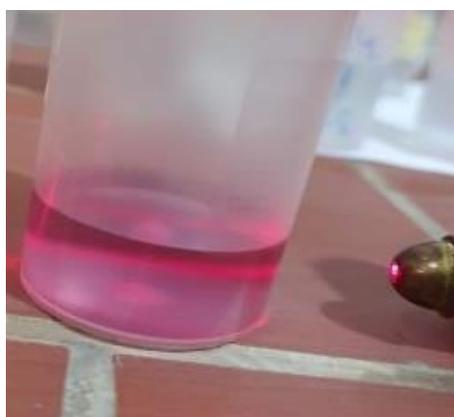
V: solution volume in Liter,

Q<sub>e</sub>: the capacity of adsorption (mg. g<sup>-1</sup>).

## **Results and discussion**

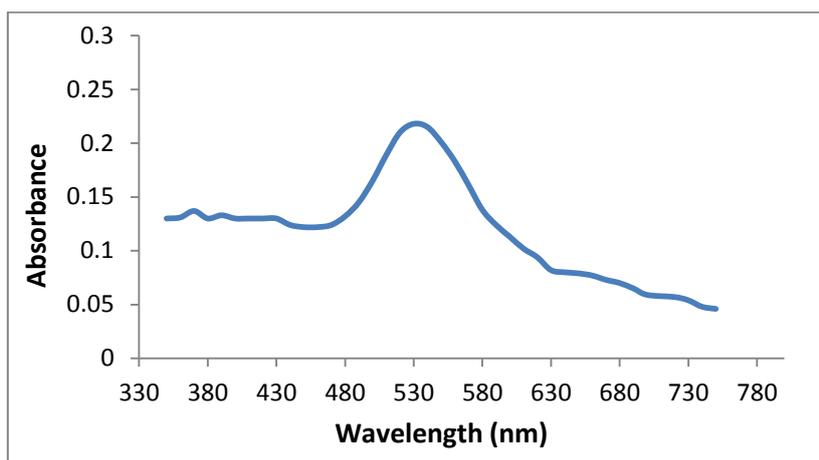
### **Characterization of gold nanoparticles**

The formation of gold nanoparticles was diagnosed by passing laser beam through the solution. The light without suffering refraction due to the occurrence of the Tyndall phenomenon hence the light scattered by fine particles in the solution of nanoparticles. Fig.1 shows this scattering.



**Figure 1. Shows the laser beam passing of gold nanoparticles solution.**

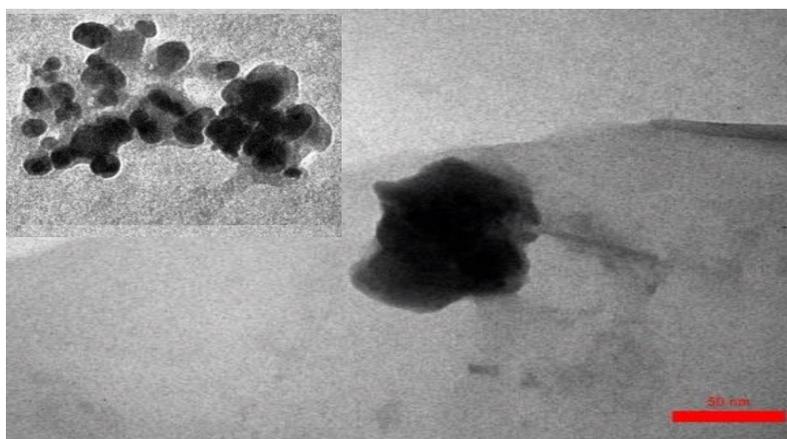
Then the plasmon resonance band for gold nanoparticles was examined by using UV-Vis spectroscopy type (U -1500, Hitachi). The plasmon resonance studied on the range between 350-750 nm. The plasmon band of electromagnetic spectrum for gold nanoparticles was in the wavelength 530 nm which is agreement with references <sup>15, 16</sup>.



**Figure 2. Uv-Vis spectra of Gold nanoparticles**

### **Transmission Electron Microscope**

The morphology of gold nanoparticles was studied using Transmission Electron Microscope TEM. It was used to identify the size and the shape for gold nanoparticles. TEM image of synthesized gold nanoparticles observed as in Fig.3.



**Figure 3. TEM image of gold nanoparticles**

## Dynamic light scattering (DLS) analysis

DLS can measure the size distribution by zeta sizer. Moreover, the stability of synthesized gold nanoparticles was evaluated with zeta potential as shown in Fig.4 and Fig.5 respectively

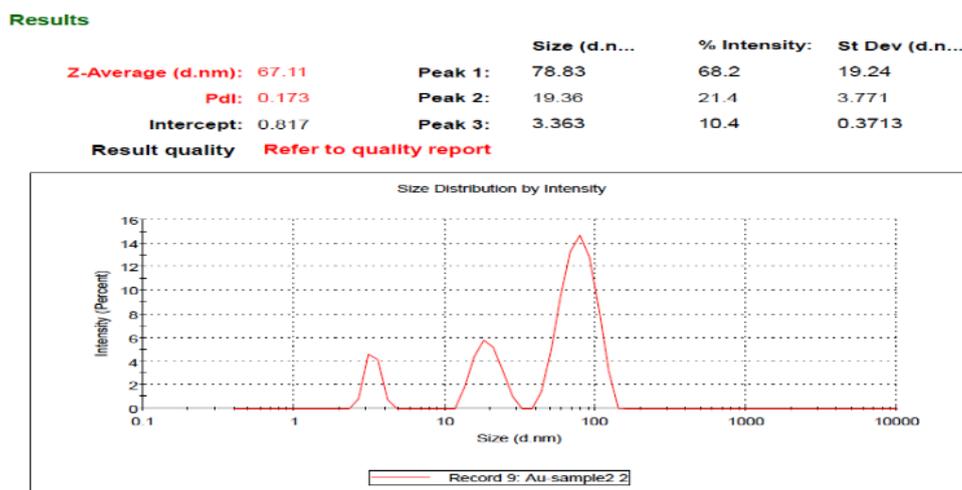


Figure 4. Shows zeta sizer of gold nanoparticles

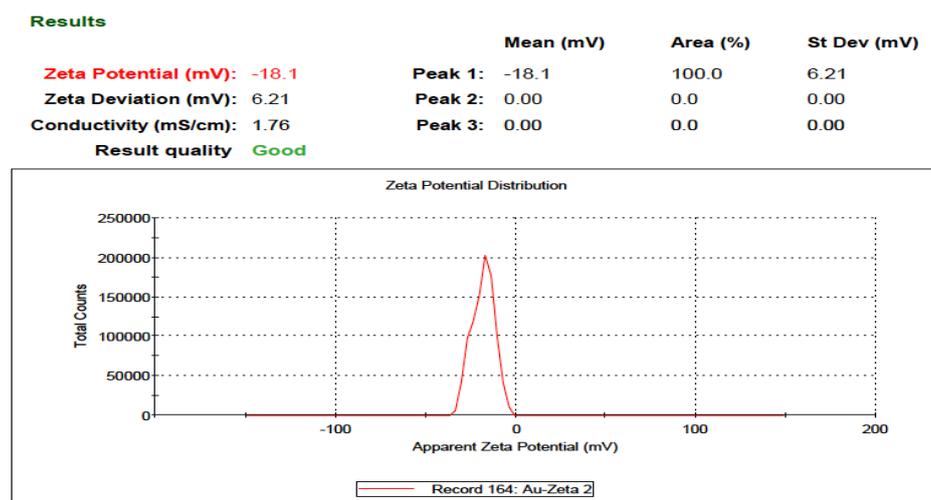
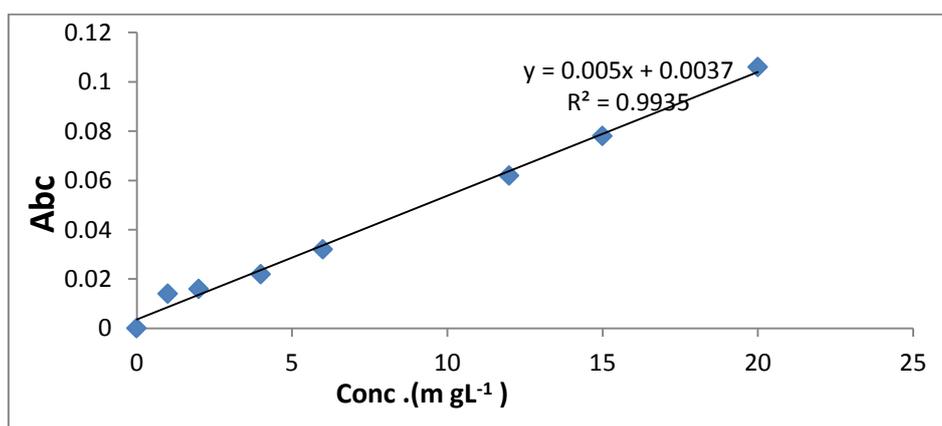


Figure 5. Shows the zeta potential of gold nanoparticles

From the figures it's clear that gold nanoparticles are nanomaterials and from Fig.5 zeta potential shows the potential for gold nanoparticles is -18.1(mV) which indicate for the stability of gold nanoparticles.

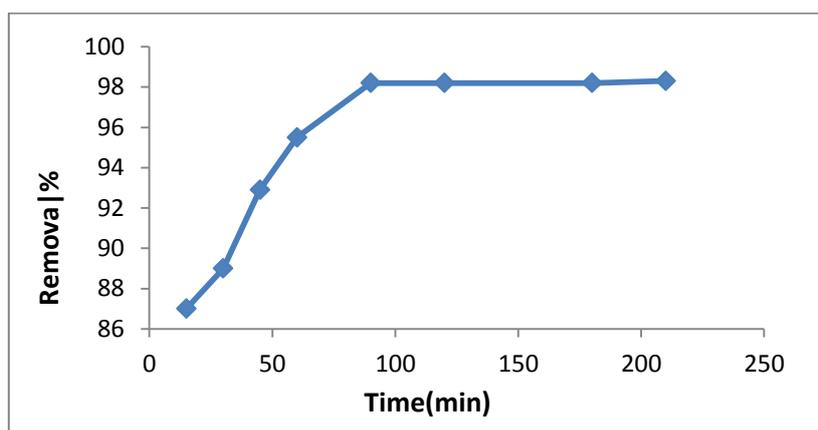
### **Study the optimum conditions for adsorption prosses**

Gold nanoparticles surface used as an adsorbent. The adsorption was studied using the batch method, several factors such as contact time, pH, the initial concentration of lead and the weight of adsorbent have been studied. First the standard curve for lead ion was measured as can be seen in Fig.6. in the range of concentration (1-20) mg.L<sup>-1</sup>. The concentration of (10 ppm) of lead was chosen for adsorption study then the Eq.1 was used for calculate the adsorption percentage of removal of lead from polluted water using gold nanoparticles as an adsorbent.



**Figure 6. The standard curve of lead ion measured by using atomic absorption spectroscopy.**

The time effect on lead ions elimination process on the gold nanoparticles surface was studied in different time periods ranging from 15-210 min at a fixed volume of gold nanoparticles 15ml and a constant concentration and volume of lead  $10\text{mg}^{-1}\text{L}^{-1}/15\text{ml}$ .

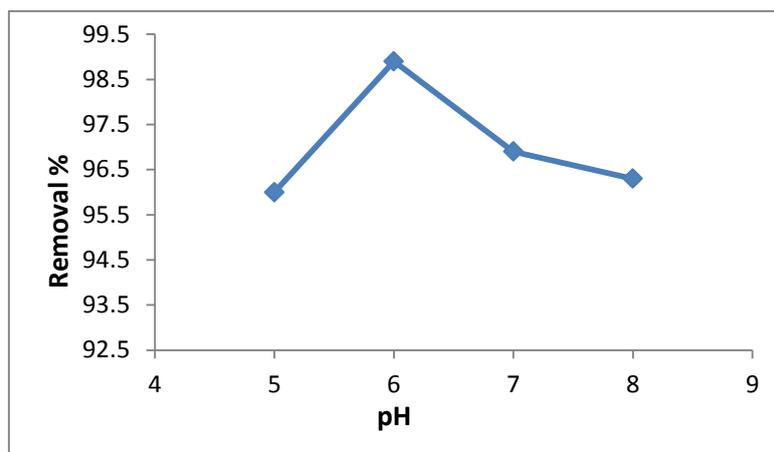


**Figure 7. The influence of contact time on lead removal by gold nanoparticles**

The results of the study showed that the time needed to reach the equilibrium state is (90min), and the percentage of removal was 98.2, as shown in the Fig. 7.

### **The influence of pH on adsorption of lead**

The adsorption of heavy metal ions from aqueous solutions affected by several factors, the most important one the acidity function which affecting due to its influence on the solubility of the metal ion. The adsorption of lead ion on the surface of gold nanoparticles in different acidic functions ranging from 4 - 8 has been studied as can be seen in Fig. 8.



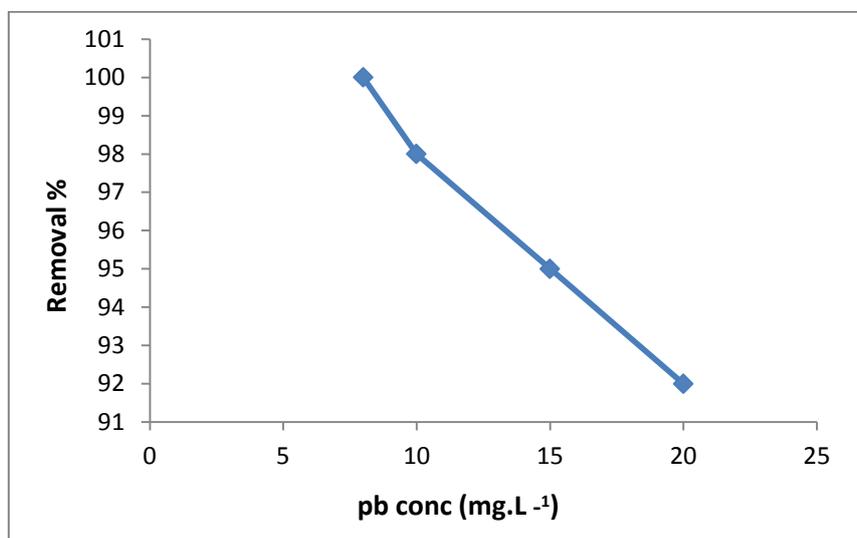
**Figure 8. The influence of pH on the lead removal by gold nanoparticles**

From the results of the study, we note that the percentage of lead ions removal using gold nanoparticles is the greatest possible adsorption percentage at the  $\text{pH} = 6$ , this is due to the fact that at low pH values, there is an abundance of hydrogen ions ( $\text{H}^+$ ) that contest with lead ions on the active sites on the gold nanoparticles surface, so the percentage of adsorption decreases<sup>17</sup>.

### **The influence of initial metal concentration**

The lead ions adsorption on the surface of gold nanoparticles was carried out using variable initial concentrations of lead ion solution 8,10,15,20  $\text{mg} \cdot \text{L}^{-1}$  at  $25^\circ\text{C}$  and  $\text{pH} = 6$ . It is clear from the results that the percentage of removal increases by decreasing the initial concentration of the lead ions under study. The increasing in the percentage of adsorption is due to the fact that in the case of high concentrations,

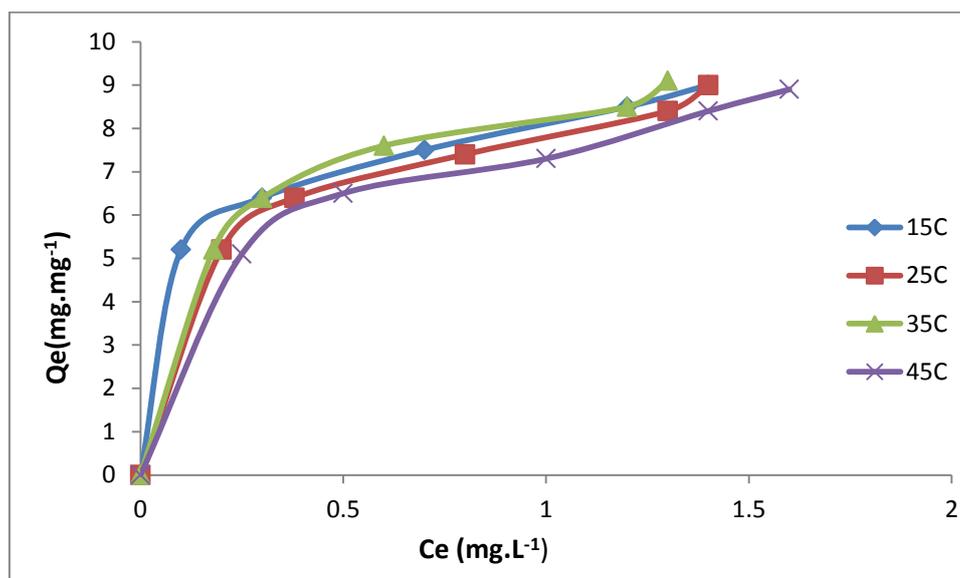
there is a relatively large number of elemental ions and thus they occupy the largest possible number of active sites on the adsorbing surface <sup>18</sup>.



**Figure 9. The influence of concentration of metal on the lead removal by gold nanoparticles.**

### **Adsorption Isotherm**

Adsorption isotherms are defined as a description of the adsorption process in equilibrium between the solution and the solid phase of the adsorbent surface, or as the relationship between the amount of an adsorbent on a surface and the concentration of an adsorbent at constant temperatures. The efficiency of removal of lead from aqueous solution on gold nanoparticles was carried out at various temperatures (15, 25, 35, 45°C) and the adsorption capacity ( $Q_e$ ) for every concentration in the equilibrium solution ( $C_e$ ) were calculated according to Eq.2. The shape of metal adsorption isotherm is shown in Fig. 11.



**Figure 10. Adsorption isotherm of lead onto gold nanoparticles at different temperatures**

The adsorption isotherm form of lead ion on the gold nanoparticles surface shows that the general form of lead ion adsorption is in great agreement with class L group 4 according to Giles classification, the orientation of the adsorbed molecules is horizontal on the surface, and the adsorption is monolayer. The results obtained from studying the temperature effect on the adsorption of lead ions Fig. 12 and Fig. 13 were used in the application of the mathematical Eq.3 and Eq.4 for the Langmuir isotherm and the Freundlich isotherm for adsorption.

$$\frac{C_e}{Q_e} = \frac{1}{Q_m b} + \frac{C_e}{Q_m} \dots 3$$

Whereas  $Q_m$  is the maximum adsorption capacity ( $\text{mg.g}^{-1}$ ) and  $b$  is Langmuir constant

$$\text{Log } Q_e = \log K_f + \frac{1}{n} \log C_e \dots 4$$

K<sub>f</sub> is the Freundlich constant and n is the Freundlich indicator.

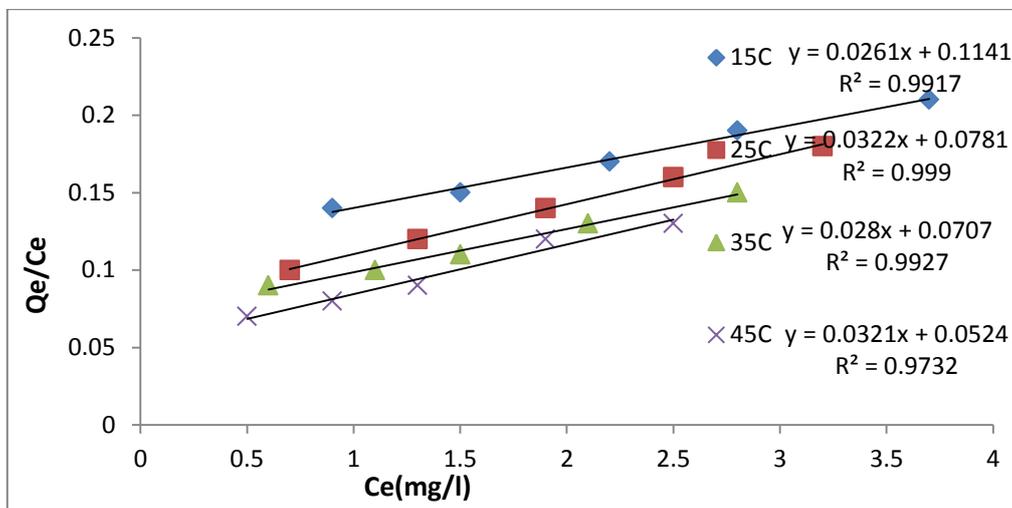


Figure 11. Linear form of Langmuir isotherm of lead on gold nanoparticles

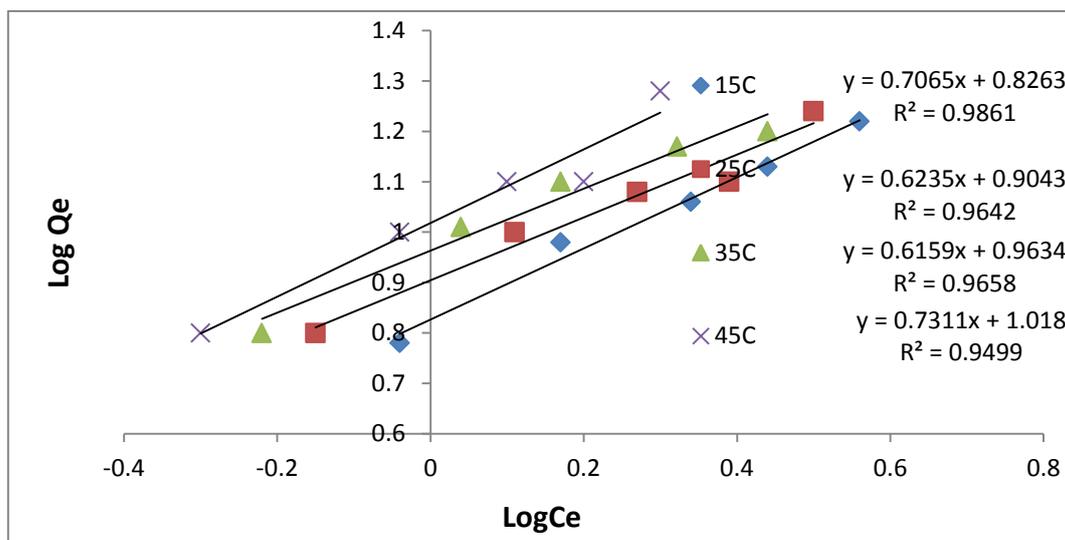


Figure 12. Linear form of Freundlich isotherm of lead on gold nanoparticles

**Table 1. Results of application of Langmuir & Freundlich isotherm on the studied system.**

	Temperature °C	Langmuir			Freundlich		
		Q <sub>m</sub> (mg/g)	b (l/g)	R <sup>2</sup>	K <sub>f</sub>	n	R <sup>2</sup>
<b>Pb(II)</b>	15	38.31	0.22	0.991	6.70	1.41	0.986
	25	31.05	0.41	0.999	8.01	1.60	0.964
	35	35.71	0.39	0.992	9.13	1.62	0.965
	45	31.15	0.61	0.973	10.2	1.36	0.949

From observing from Table 1 the values of the slope and the correlation coefficient R<sup>2</sup>, it is clear that

Langmuir equation is the most compatible with the adsorption process of the lead ions on the surface of gold nanoparticles <sup>19</sup>

### Thermodynamic Study

The temperature effect on the lead ion adsorption on the surface of gold nanoparticles was studied at different temperatures 15,25,35 and 45 °C at different concentrations. The study of the temperature effect on the removal process helps in estimating the values of thermodynamic functions such free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) for the importance of these functions in understanding the adsorption process. Eq.5 - 8 used for calculate the thermodynamic parameters:

$$\Delta G = -RT \ln K \dots 5$$

$$K = \frac{C_{solid}}{C_{liquid}} \dots\dots 6$$

$$\Delta G = \Delta H - T\Delta S \dots 7$$

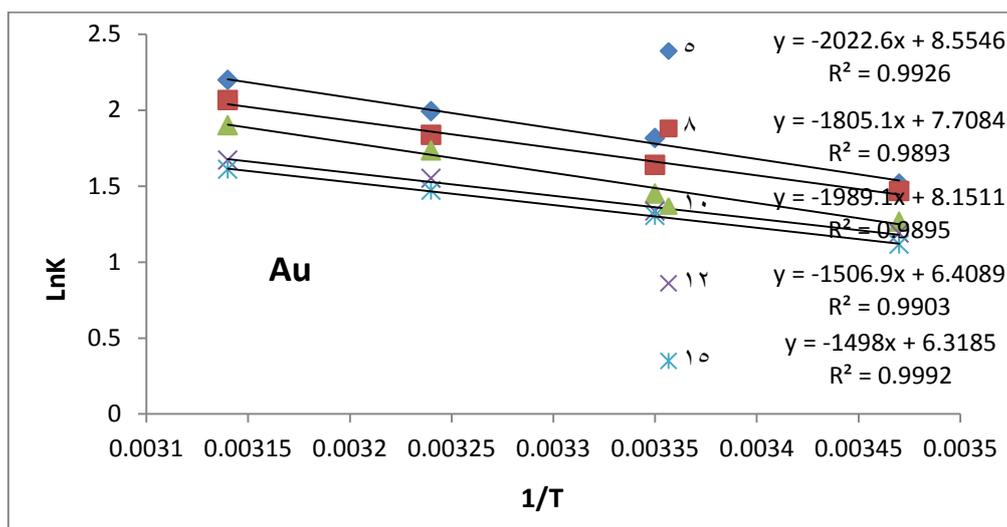
$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \dots 8$$

Where  $\Delta G$  is the change in free energy ( $\text{KJ.mol}^{-1}$ ),  $K$  is The thermodynamic equilibrium constant for the adsorption process,  $C_{solid}$  is the solid phase concentration at equilibrium ( $\text{mg/l}$ ),  $C_{liquid}$  is the liquid phase concentration at equilibrium ( $\text{mg/l}$ ),  $R$  is the gas constant ( $0.008314 \text{ KJ. mol}^{-1} . \text{K}^{-1}$ ). and  $T$  is the temperature in Kelvin.

**Table 2. Thermodynamic function for adsorption of pb (II).**

	$C_0$ ( $\text{mg L}^{-1}$ )	Equilibrium constant $K$				$-\Delta G \text{ KJ.mol}^{-1}$				$-\Delta H$ $\text{K.J.mol}^{-1}$	$\Delta S$ $\text{J.mol. K}^{-1}$
		Temperature $^{\circ}\text{C}$				Temperature $^{\circ}\text{C}$					
		15	25	35	45	15	25	35	45		
<b>Pb(II)</b>	5	4.55	6.14	7.33	9.00	3.63	4.34	5.05	5.76	16.81	0.071
	8	4.33	5.15	6.27	7.88	3.42	4.06	4.70	5.34	15.00	0.064
	10	3.54	4.26	5.66	6.69	2.75	3.42	4.09	4.76	16.53	0.067
	12	3.28	3.80	4.71	5.31	2.73	3.26	3.79	4.32	12.52	0.053
	15	3.05	3.68	4.35	5.00	2.52	3.04	3.56	4.08	12.45	0.052

The thermodynamic variables were calculated through Eq. 8, where the values in Table 2 were calculated based on the Van, t Hoff - Arinos equation, depending on the Fig. 14 where the slope is  $(-H/R)$  and the cut-off is  $(\Delta S/R)$  <sup>20</sup>.



**Figure 13.** Plot of van't Hoff relationship between  $\ln K$  and  $1/T$ .

From the results shown in Table 2, it is clear that values of the adsorption temperature ( $\Delta H$ ) of the lead ion on the gold nanoparticles surface are positive values, meaning that the adsorption process is an endothermic type. The positive values of  $\Delta S$  indicate that the adsorbed particles are less uniform (a random reaction) while the negative  $\Delta G$  values indicate that the adsorption process of the lead ion on the gold nanoparticles surface is a spontaneous process<sup>21,22</sup>

### Kinetics of adsorption

The kinetic adsorption data of lead ion on gold nanoparticles were studied using two kinetic models: False first order and pseudo second order. Eq.9 shows the kinetic model according to the false first-order equation.

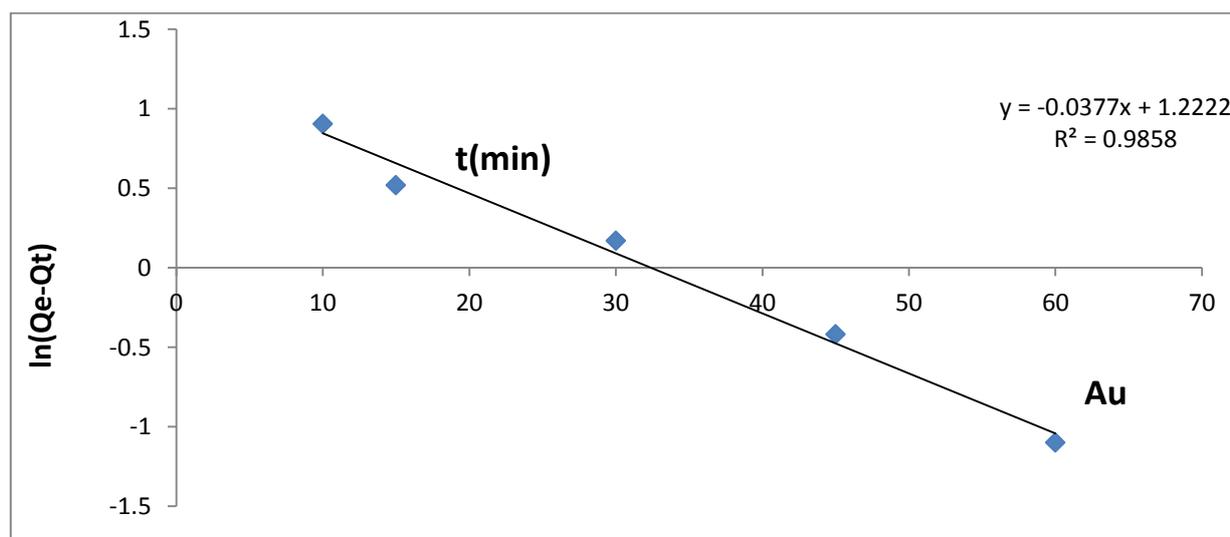
$$\ln(Q_e - Q_t) = \ln Q_e - K_1 t \dots 9$$

Whereas  $Q_e$  and  $Q_t$  the capacity of adsorption at equilibrium and time  $t$  respectively and  $(k_1)$  ( $\text{min}^{-1}$ ) is the adsorption rate constant for the first order

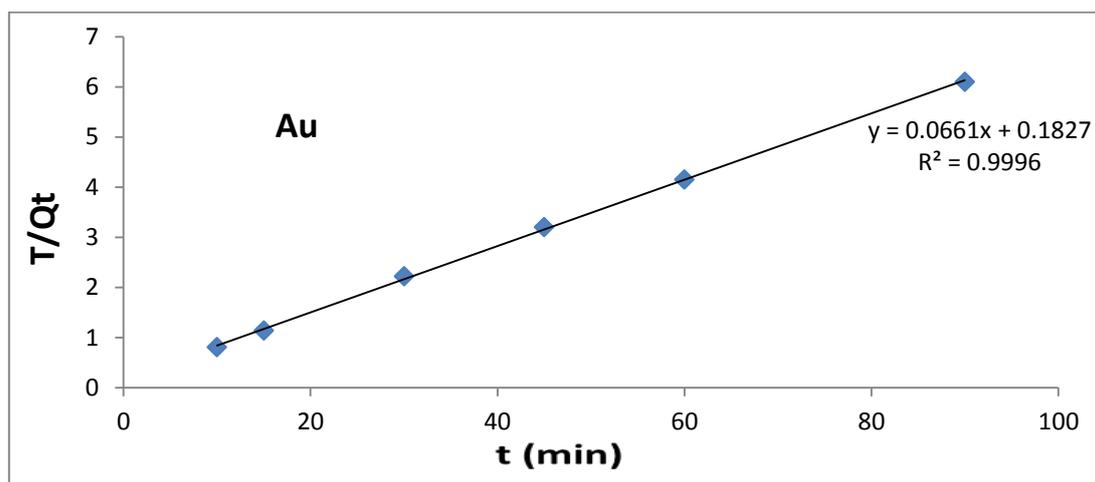
And  $t$  is the contact time (min). The obtained results were used in the application of the pseudo-first-order adsorption kinetics equation and Table 3 shows the values of  $\ln(Q_e - Q_t)$  versus time  $t$ ). Fig. 15 illustrates the linear relationship drawing of the pseudo-first-order. From the data, the value of the equilibrium rate constant  $K_1$  was calculated. And the calculated weight capacity  $Q_e(\text{cal.})$  at equilibrium obtained from the slope and intercept values of the linear relationship of the pseudo-first-order equation. The second order equation model for adsorption kinetics is expressed as:

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{1}{Q_e} t \dots 10$$

Where  $K_2$  ( $\text{g mg}^{-1} \text{min}^{-1}$ ) represents the adsorption rate of the pseudo-second-order equation. The experimental data was also applied to the pseudo-second-order adsorption kinetics equation and the Table 3 shows the values of  $(t/Q_t)$  versus time  $t$ ). The values of the adsorption rate constant for the pseudo-second-order equation  $K_2$ ) its units ( $\text{g/mg min}$ ) and adsorption capacity at equilibrium ( $Q_e$ ) units ( $\text{mg/g}$ ) were obtained from the slope and intercept value and show in Fig. 16.



**Figure 15. Linear form of Pseudo-First-Order (Lagergren) Kinetic model of lead on Au-nanoparticles**



**Figure 14. Linear form of Pseudo-Second-Order Kinetic model of lead on Au-nanoparticles**

We note through the results shown in Table 3 that the values of the experimental adsorption capacity  $Q_e$  (exp.) and the calculated adsorption capacity  $Q_e$  (cal.) at equilibrium for the pseudo-second-order equation are close to each other, so the adsorption kinetics of lead ion on the surface of gold nanoparticles follows the pseudo-second-order equation. This agrees with references<sup>23-25</sup>.

**Table 3. First order and Second order kinetic rate constant, calculated and experimental  $Q_e$  values and  $R^2$ .**

Pb(II)	Pseudo-First-Order				Pseudo-Second-Order			
	$Q_e$ exp. mg/g	$Q_e$ Cal. mg/g	K1 min <sup>-1</sup>	R2	$Q_e$ exp. mg/g	$Q_e$ Cal. mg/g	K2 (g/mg min)	R2
	14.73	3.393	0.0377	0.985	14.73	15.12	0.023	0.999

## **Conclusion**

Gold nanoparticles were prepared by wet chemistry method and were tested as an adsorbent material to remove heavy metals such lead from water. It showed a high efficiency of removal, as the percentage of lead ion removal from water reached 98%. Several techniques were used to diagnose the prepared gold particles, including TEM, DLS, which were used to prove the nanostructure for the prepared particles. The adsorption process was carried out by batch method and the results showed the dependence of the adsorption process on some factors such as equilibrium time, temperature, acidity function and initial concentration of lead ion. The results also showed that the process of removing lead ion from water was of the endothermic type, and that the adsorption process was spontaneous through negative  $\Delta G$  values and the adsorption process follows the adsorption Langmuir equation.

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### دراسة استخدام جسيمات الذهب النانوية كمادة مازة لمعالجة المياه

<sup>1</sup>ميثاق ابراهيم عبود و زينب طه ياسين العبد الله<sup>2</sup>

قسم الكيمياء- كلية التربية للعلوم الصرفة -جامعة البصرة -البصرة -العراق

#### الخلاصة

في هذه الدراسة حضرت جسيمات الذهب النانوية باستخدام الطريقة الرطبة ومن ثم استعمالها كمادة مازة لإزالة ايونات الرصاص من المياه الملوثة . حضرت جسيمات الذهب النانوية باستخدام ملح الذهب  $\text{HAuCl}_4$  كمصدر للذهب وباستخدام سكر الكلوكوز كعامل مختزل وثلاثي سترات الصوديوم كمادة مانعة للتكتل . تم تشخيص الجسيمات المحضرة باستخدام المجهر الالكتروني النافذ حيث اظهرت صورة المجهر الالكتروني ان حجم جسيمات الذهب 50 نانومتر ولمعرفة حجم واستقرارية الجسيمات المحضرة استخدمت تقنية تشتت الضوء الديناميكية كذلك تم دراسة مختلف العوامل المؤثرة على عملية الامتزاز مثل زمن الاتزان ،الدالة الحامضية والتركيز الابتدائي لفلز الرصاص .من خلال النتائج وجد ان النسبة المئوية للامتزاز تزداد بزيادة درجة الحرارة

اي ان عملية الامتزاز ماصة للحرارة كما بينت الدراسة الترموديناميكية ان عملية امتزاز ايونات الرصاص كانت تلقائية من قيم  $(\Delta G)$  السالبة كما دلت القيم الموجبة ل  $(\Delta S)$  على زيادة العشوائية لعملية الازالة. كذلك درست ايزوثيرمات الامتزاز لانكماير وفريندليش وكانت معادلة لانكماير هي الاكثر انطباقا على النظام المدروس وان حركية الامتزاز تتبع معادلة المرتبة الثانية الكاذبة .

**الكلمات المفتاحية:** الامتزاز ، جسيمات الذهب النانوية ، دراسات الترموديناميك، معالجة المياه