

# Supporting Information

## **Adsorption of Pd (II) and Au (III) Ions by Commercial Tris(2-Aminoethyl) Amine Polystyrene Polymer Beads**

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## 1. Speciation of Pd (II) and Au (III) ions

To simulate the metal ion speciation under optimum conditions calculated with Hydra and Medusa [1] software, and results shown in Figure S1.

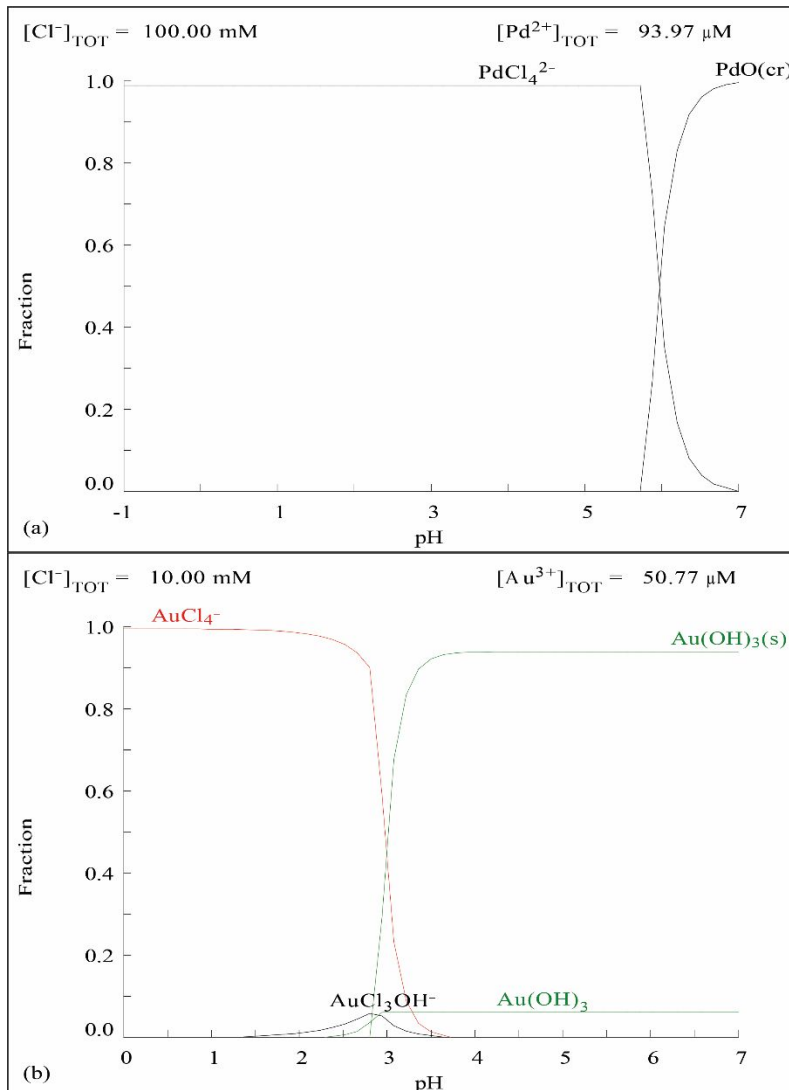


Figure S1. Pd (II) and Au (III) speciation fractions under 0.1 M and 0.01 M HCl concentrations, respectively.

## 2. Adsorption Isotherms

In order to determine which isotherm equation and its theory fitted the adsorption equilibrium state, Langmuir [2], Modified Langmuir [3], Freundlich [4], Temkin [5], Dubinin–Radushkevich [6,7], and Redlich–Peterson [8] were selected to explicate

precious metal ions–TRIS beads interactions. These isotherms and its linear forms can be seen at Table S1.

The Langmuir equation initially derived from kinetic studies has been based on the assumption that on the adsorbent surface there is a definite and energetically equivalent number of adsorption sites. The bonding to the adsorption sites can be either chemical or physical, but it must be sufficiently strong to prevent displacement of adsorbed molecules along the surface. Thus, localised adsorption was assumed as being distinct from non-localised adsorption, where the adsorbed molecules can move along the surface. Because the bulk phase is constituted by a perfect gas, lateral interactions among the adsorbate molecules were neglected. On the energetically homogeneous surface of the adsorbent a monolayer surface phase is thus formed. Langmuir, for the first time, introduced a clear concept of the monomolecular adsorption on energetically homogeneous surfaces [2,9].

The  $K_L$  and  $K_{ML}$  are the Langmuir isotherm constants. In this study, unit of  $K_L$  is L/mg and  $K_{ML}$  is dimensionless constants. Due to the Langmuir equilibrium constant,  $K_L$ , is not being dimensionless, it is not theoretically suitable for using in thermodynamic calculations. Instead, the Modified Langmuir isotherm has been proposed and the equilibrium constant is unitless [3]. The theoretical monolayer saturation capacity,  $q_m$ , dimension is given as mg/g.

**Table S1.** Adsorption isotherms and its linear forms.

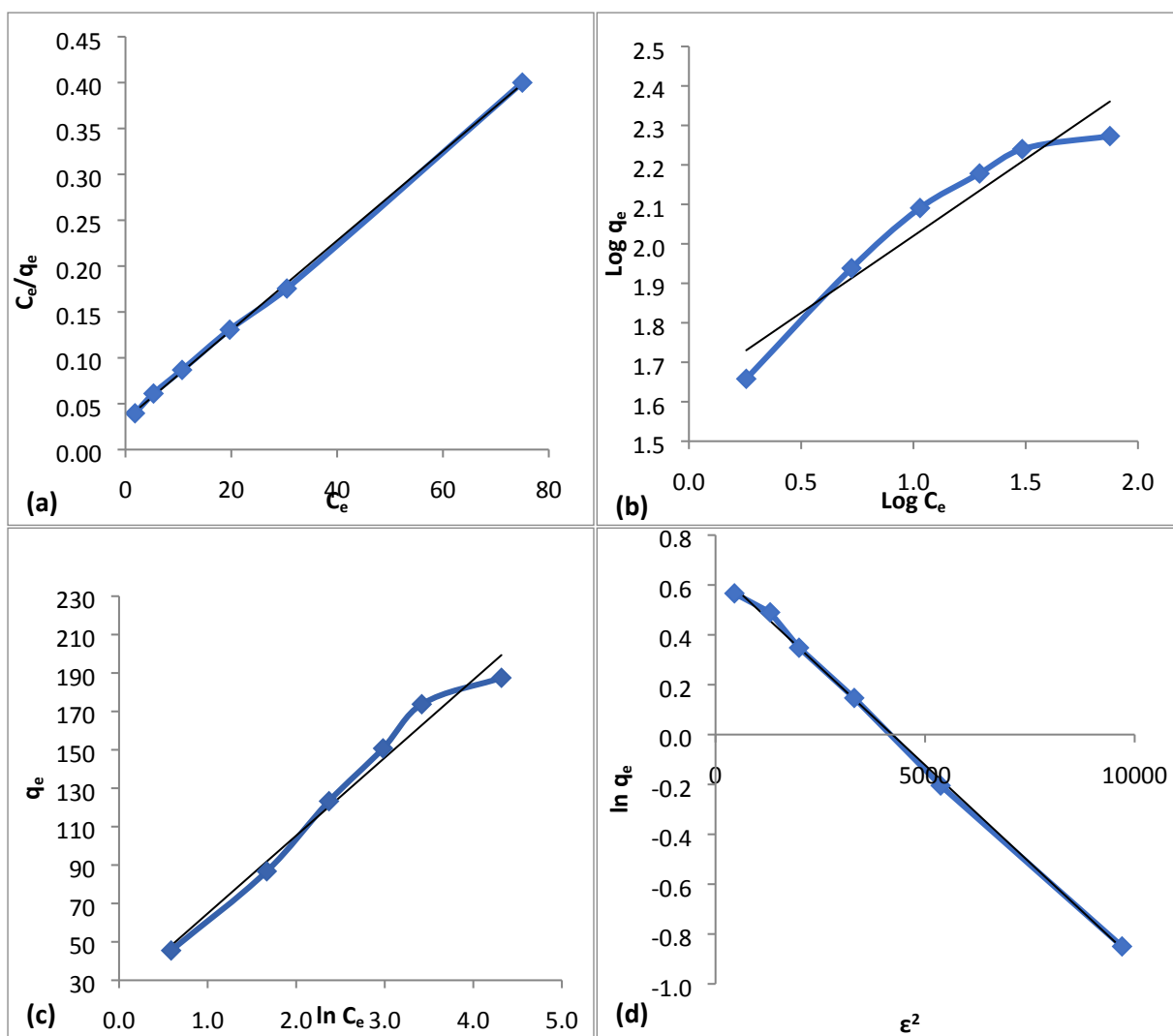
Isotherm	Linear Form	X & Y	Slope & cut-off point
Langmuir [10,11]	$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m}$	$\tan \alpha = \frac{1}{q_m}$ $cutoff = \frac{1}{K_L q_m}$

Modified Langmuir [3]	$q_e = \frac{q_m K_{ML} C_e}{(C_s - C_e) + K_{ML} C_e}$	$\frac{C_e}{q_e} = \frac{C_s}{q_m K_{ML}} + \frac{(K_{ML} - 1) C_e}{K_{ML} q_m}$		$\tan \alpha = \frac{(K_{ML} - 1) C_s}{K_{ML} q_m}$ $cutoff = \frac{C_s}{q_m K_{ML}}$
Freundlich [4]	$q_e = K_f C_e^{1/n}$	$\log q_e = -\log K_f + \frac{1}{n} \log C_e$	$x = \log C_e$ $y = \log q_e$	$\tan \alpha = \frac{1}{n}$ $cutoff = -\log K_f$
Temkin [5]	$q_e = \frac{RT}{b} \ln (AC_e),$ $RT/b = B$	$q_e = B \ln A + B \ln C_e$	$x = \ln C_e$ $y = q_e$	$\tan \alpha = B$ $cutoff = B \ln A$
(D-R) [6,7]	$q_e = q_m e^{-\beta \varepsilon^2}$ $\varepsilon = RT \left(1 + \frac{1}{C_e}\right)$	$\ln q_e = \ln q_m - \beta \varepsilon^2$	$x = \varepsilon^2$ $y = \ln q_e$	$\tan \alpha = \beta$ $cutoff = q_m$
(R-P) [8]	$q_e = \frac{AC_e}{1 + BC_e^g}$	$\ln \left( \frac{C_e}{q_e} - 1 \right) = g \ln (C_e) + \ln (B)$	-	-

The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant called separation factor ( $R_L$ ) which is defined by the following equation

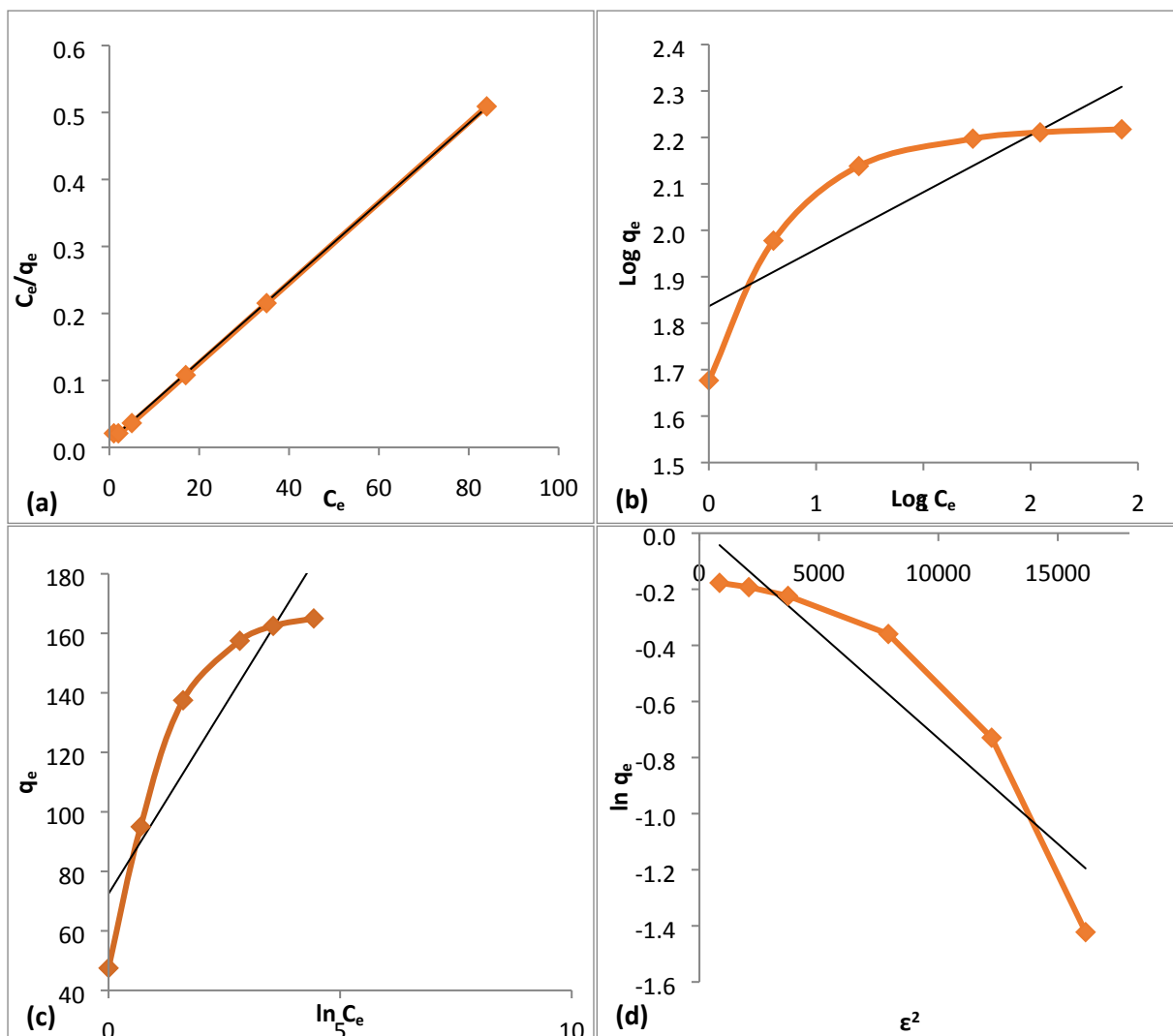
$$R_L = \frac{1}{1 + K_L C_0} \quad (1)$$

where  $C_0$  (mg/L) is the initial metal ion concentration and  $K_L$  (L/mg) is the Langmuir constant related to the energy of adsorption. In this context, the value of  $R_L$  indicates the shape of the isotherms to be either unfavorable ( $R_L > 1$ ), linear ( $R_L = 1$ ), favorable ( $0 < R_L < 1$ ) or irreversible ( $R_L = 0$ ) [12,13].



**Figure S2.** The Langmuir (a), Freundlich (b), Temkin (c), Dubinin–Radushkevich (d) isotherm regression graphics, parameters and  $R^2$  values for Pd (II) adsorption onto TRIS beads.

The Freundlich isotherm is an empirical equation employed to describe heterogeneous systems and equation shown in Table S1. In this equation,  $K_f$ , ( $mg^{1-1/n}L^{1/n}g^{-1}$ ) is the Freundlich constant related to the bonding energy, and  $n$ , ( $g/L$ ) is the heterogeneity factor. The slope ( $1/n$ ) ranges between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, and it becomes more heterogeneous when its value gets closer to zero. Whereas, a value below unity implies chemisorptions process where  $1/n$  above one is an indicative of cooperative adsorption [12,14].



**Figure S3.** The Langmuir (a), Freundlich (b), Temkin (c), Dubinin–Radushkevich (d) isotherm regression graphics, parameters and  $R^2$  values for Au (III) adsorption onto TRIS beads.

By ignoring the extremely low and large value of concentrations, the derivation of the Temkin isotherm assumes that the fall in the heat of sorption is linear rather than logarithmic. Temkin equation is excellent for predicting the gas phase equilibrium, conversely complex adsorption systems including the liquid-phase adsorption isotherms are usually not appropriate to be represented [15]. In this equation,  $A$  (L/mg) is the equilibrium binding constant corresponding to the maximum binding energy,  $b$  (J/mol) is Temkin isotherm constant and constant  $B$  (dimensionless) is related to the heat of adsorption.

Radushkevich [16] and Dubinin [17] have reported that the characteristic sorption curve is related to the porous structure of the sorbent (Table S1). Where the constant,  $\beta$ , ( $\text{mmol}^2/\text{J}^2$ ) is D-R constant related to the mean free energy of sorption per mole of the sorbate as it is transferred to the surface of the solid from infinite distance in the solution and can be correlated using following relationship

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

and  $q_m$ , (mmol/g) is denoted as the single layer capacity. In a deeper explanation,  $E$  value indicates the mechanism of the adsorption reaction. When  $E < 8 \text{ kJ/mol}$ , physical forces may affect the adsorption. If  $E$  is  $8 < E < 16 \text{ kJ/mol}$ , adsorption is governed by ion exchange mechanism, while for the values of  $E > 18 \text{ kJ/mol}$ , adsorption may be dominated by particle diffusion [18]. The model has often successfully fitted high solute activities and the intermediate range of concentrations data well, but has unsatisfactory asymptotic properties and does not predict the Henry's law at low pressure [12,18]. Meanwhile, the parameter  $\varepsilon$  known as Polanyi potential and can be correlated as

$$\varepsilon = RT \left( 1 + \frac{1}{C_e} \right) \quad (3)$$

where  $R$ ,  $T$  and  $C_e$  represent the gas constant (8.314 J/mol K), absolute temperature (K) and adsorbate equilibrium concentration (mg/L), respectively.

The Redlich-Peterson isotherm contains three parameters ( $A(\text{L/g})$ ,  $B(\text{L/mg}^{1-1/A})$ ,  $g$ ) and incorporates the features of the Langmuir and the Freundlich isotherms [8]. Its equation and linear form can be seen in Table 1. Isotherm unitless constants  $g$  is gives between  $0 < g < 1$  values. When  $g = 1$ , adsorption isotherm fits Langmuir isotherm. If  $g = 0$ , isotherm is now fully Freundlich isotherm [19,20]. Values between this concerned for isotherm

representation. Due to having this versatility, it can be applied either in homogeneous or heterogeneous systems.

In this study two error functions, the coefficient of determination and nonlinear chi-square test have been used for analyzing the adsorption system. Coefficient of determination is defined as [18]

$$r^2 = \frac{\sum(q_{e,meas} - \overline{q_{e,calc}})^2}{\sum(q_{e,meas} - \overline{q_{e,calc}})^2 + (q_{e,meas} - q_{e,calc})^2} \quad (4)$$

where  $q_{e,meas}$  (mg/g) is the amount of dye exchanged by the surface of adsorbent obtained from experiment,  $q_{e,calc}$  the amount of adsorbate obtained by isotherm models and  $\overline{q_{e,calc}}$  the average of  $q_{e,calc}$  (mg/g).

Nonlinear chi-square test is a statistical tool necessary for the best fit of an adsorption system. It is expressed as

$$X^2 = \sum_{i=1}^n \frac{(q_{e,calc} - q_{e,meas})^2}{q_{e,meas}} \quad (5)$$

### 3. Adsorption kinetics

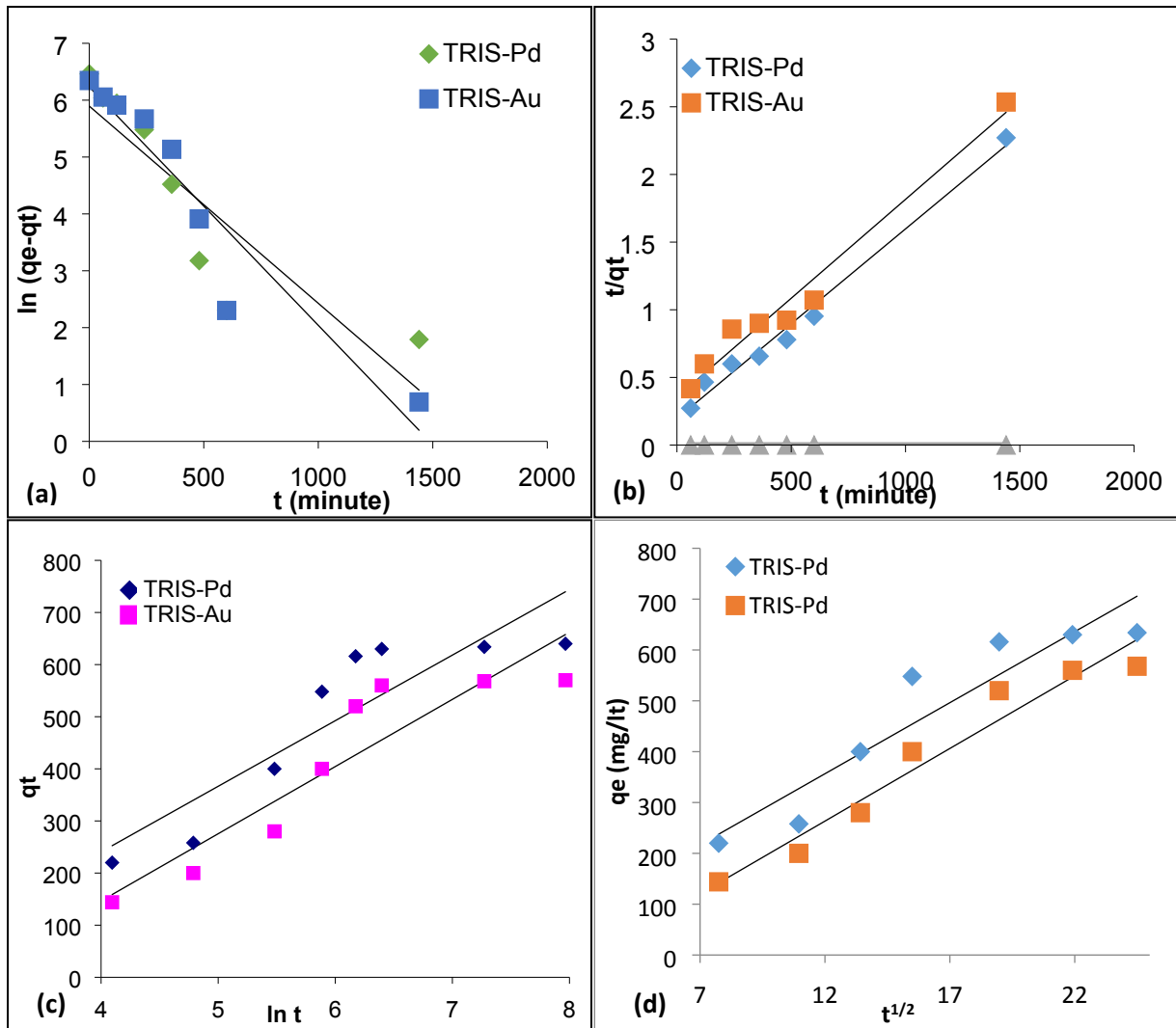
Adsorption kinetics that is examining the change of adsorption on solid surfaces from solutions up to reaching equilibrium, a suitable kinetic model is needed to analyze the rate data. With the development of the adsorption equilibrium theory on heterogeneous solid surfaces, the development of the kinetic adsorption-desorption theory has also progressed [14]. In order to understand best fitting the kinetic theory and its equation of studied metal ion adsorption kinetics, the pseudo first and pseudo second order, Elovich, and intraparticle diffusion equations were applied. These isotherms, linear forms, and their constants can be seen at Table 2.



**Table S2.** Used adsorption kinetic equations and its linear forms.

	<b>Equation</b>	<b>Linear Form</b>	<b>Constants</b>
<b>Lagergren first-order</b> [21]	$\frac{dq_t}{dt} = k_1(q_e - q_t)$	$\log(q_e - q_t) = \log q_e - \frac{k_1}{2,303}t$	$k_1 (\text{min.}^{-1})$
<b>Pseudo second-order</b> [22]	$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$	$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t$	$k_2(\text{g.mg}^{-1}.\text{min.}^{-1})$
<b>Elovich</b> [23,24]	$\frac{dq_t}{dt} = \alpha e^{-\beta q_t}$	$q_t = \frac{1}{\beta} \ln(\alpha \beta) + \frac{1}{\beta} \ln t$	$\alpha (\text{mg.g}^{-1}.\text{min.}^{-1})$ $\beta (\text{mg.g}^{-1})$
<b>Intraparticle diffusion</b> [18]	$q_t = k_{int}t^{1/2}$		$k_{int} (\text{mg.g}^{-1}.\text{min.}^{-\frac{1}{2}})$

An adsorption-desorption kinetics can include the following four steps: external diffusion from solution to interface, diffusion into pores, diffusion of molecules on surface, and realization of adsorption-desorption process [14,25]. Integrated linear pseudo-first and pseudo-second order, Elovich, and intraparticle diffusion kinetic equation form function graphics and their  $R^2$  values can be seen in **Figure S4**.



**Figure S4.** Pseudo-first (a), pseudo-second (b) order, Elovich (c), and intraparticle diffusion (d) kinetic equation parameters and  $R^2$  values.

#### 4. Adsorption Thermodynamic

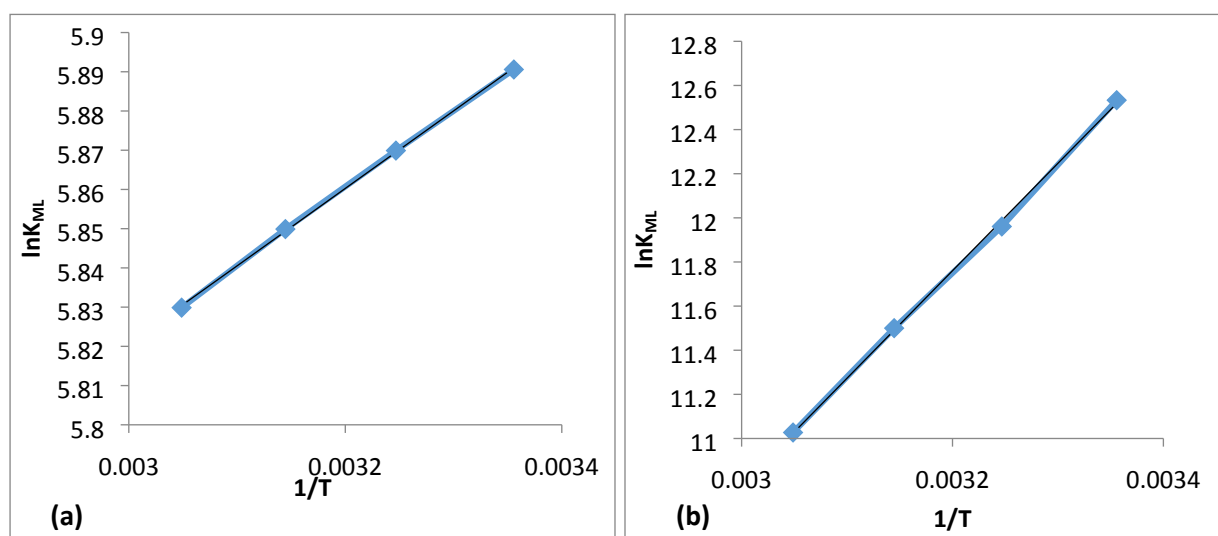
Thermodynamic parameters such as Gibb's free energy ( $\Delta G^0$ ), enthalpy change ( $\Delta H^0$ ) and change in entropy ( $\Delta S^0$ ) for the adsorption of metal ions on TRIS polymer beads have been determined by using the following equations

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (6)$$

$$\Delta G^0 = -RT \ln(K_{ML}) \quad (7)$$

$$\log(K_{ML}) = \frac{\Delta S^0}{2.303 R} - \frac{\Delta H^0}{2.303 RT} \quad (8)$$

where  $q_e$  is the amount of metal ion adsorbed per unit mass of beads (mg/g),  $C_e$  is equilibrium concentration (mg/L) and  $T$  is temperature in K and  $R$  is the gas constant (8.314 J/molK).  $K_{LM}$  is dimensionless Modified Langmuir constant at each temperature. It is calculated from linear regression solve of isotherm equation. Considering the relationship between  $\Delta G^0$  and  $K_L$ ,  $\Delta H^0$  and  $\Delta S^0$  were determined from the slope and intercept of the van't Hoff plots of  $\log(K_{ML})$  versus  $1/T$ . Negative values of  $\Delta G^0$  confirm the feasibility of the process and the spontaneous nature of the adsorption [18]. In general, the  $\Delta H^0$  value of physisorption is smaller than 40 kJ/mol. The positive value of  $\Delta H^0$  is indicating that the adsorption reaction was endothermic [25]. The negative entropy change ( $\Delta S^0$ ) for the process was caused by the decrease in degree of freedom of the adsorbed species [15].



**Figure S5.** van't Hoff plot of  $\ln K_d$  versus  $1/T$  for the estimation of thermodynamic parameters of Pd (II) (a), and Au (III) (b) adsorption.

## 5. SEM Image of TRIS Polymer Beads

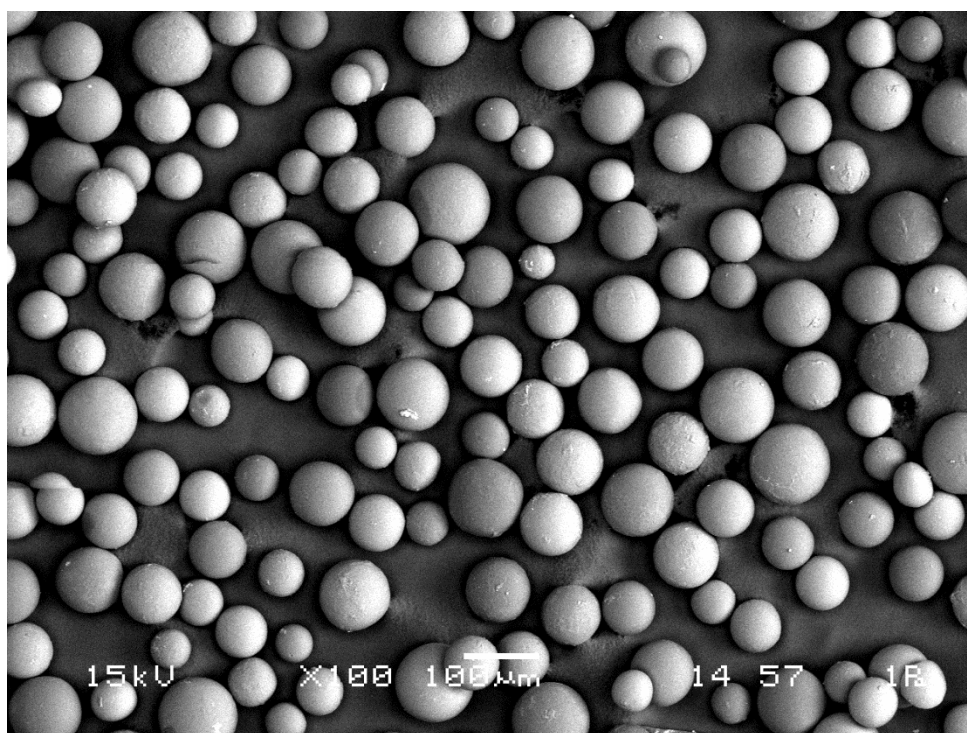


Figure S6. SEM image of TRIS polymer beads.

## 6. Experimental Result Tables

**Table S3.** Experimental result of effecting [HCl] on sorption of Pd (II) and Au (III) onto TRIS polymer beads ( $C_0=10$  mg/L, 293 K,  $m=10$  mg,  $V=50$  ml, 24 hours).

Pd (II) adsorption, Parameters	First experiment		Second experiment		Avarage $q_e$ (mg/g)	Standard deviation
	$C_e$ (mg/L)	$q_e$ (mg/g)	$C_e$ (mg/L)	$q_e$ (mg/g)		
3 M HCl	9.5	2.5	8.7	6.5	4.5	2.8
1 M HCl	4.8	26	6.4	18	22.0	5.7
0.1 M HCl	1.6	42	0.4	48	45.0	4.2
0.01 M HCl	6.5	17.5	8.7	6.5	12.0	7.8
Au (III) adsorption, Parameters	First experiment		Second experiment		Avarage $q_e$ (mg/g)	Standard deviation
	$C_e$ (mg/L)	$q_e$ (mg/g)	$C_e$ (mg/L)	$q_e$ (mg/g)		
3 M HCl	4	30	4.8	26	28.0	2.8
1 M HCl	1.9	40.5	3.9	30.5	35.5	7.1
0.1 M HCl	1	45	1.8	41	43.0	2.8
0.01 M HCl	0.2	49	0.6	47	48.0	1.4

**Table S4.** Experimental result of the effects of initial metal ion concentrations to adsorption in Pd (II) onto TRIS beads. ( $C_0=10-150$  mg/L, 10mg/50ml TRIS polymers, [HCl]=0.1, 24 hours).

Pd (II), $C_0$ , 298K	First experiment		Second experiment		Avarage $q_e$ (mg/g)	Standard deviation
	$C_e$ (mg/L)	$q_e$ (mg/g)	$C_e$ (mg/L)	$q_e$ (mg/g)		
20	4.1	39.75	3.5	41.25	3.8	1.1
40	5.1	87.25	5.4	86.5	5.3	0.5
60	5	137.5	5.6	136	5.3	1.1
80	7.9	180.25	7.5	181.25	7.7	0.7
100	24.3	189.25	28.7	178.25	26.5	7.8
150	77.1	182.25	72.9	192.75	75.0	7.4

<b>Pd (II), C<sub>0</sub>, 328K</b>	<b>First experiment</b>		<b>Second experiment</b>		<b>Avarage q<sub>e</sub> (mg/g)</b>	<b>Standard deviation</b>
	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>		
20	2.1	44.8	2.3	44.3	2.2	0.4
40	6.0	85.0	5.2	87.0	5.6	1.4
60	11.3	121.8	12.7	118.3	12.0	2.5
80	23.0	142.5	19.2	152.0	21.1	6.7
100	33.9	165.4	33.8	165.6	33.8	0.1
150	78.2	179.5	79.0	177.5	78.6	1.4

**Table S5.** Experimental result of the effects of initial metal ion concentrations to adsorption in Au (II) onto TRIS beads. (C<sub>0</sub>=10-150 mg/L, 10mg/50ml TRIS polymers, [HCl]=0.01, 24 hours).

<b>Au (III), C<sub>0</sub>, 298K</b>	<b>First experiment</b>		<b>Second experiment</b>		<b>Avarage q<sub>e</sub> (mg/g)</b>	<b>Standard deviation</b>
	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>		
20	0.5	48.8	1.5	46.3	47.5	1.8
40	1.1	97.3	2.9	92.8	95.0	3.2
60	4.7	138.3	5.3	136.8	137.5	1.1
80	18.0	155.0	16.0	160.0	157.5	3.5
100	33.9	165.3	36.1	159.8	162.5	3.9
150	85.9	160.3	82.1	169.8	165.0	6.7

<b>Au (III), C<sub>0</sub>, 328K</b>	<b>First experiment</b>		<b>Second experiment</b>		<b>Avarage q<sub>e</sub> (mg/g)</b>	<b>Standard deviation</b>
	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>		
20	1.9	45.4	2.6	43.6	44.5	1.2
40	6.3	84.3	4.9	87.8	86.0	2.5
60	13.0	117.5	11.0	122.5	120.0	3.5
80	20.9	147.8	21.3	146.8	147.3	0.7
100	33.3	166.8	34.2	164.5	165.6	1.6
150	80.0	175.1	77.3	181.9	178.5	4.8

**Table S6.** The effects of contact time datas to Pd (II) and Au (III) adsorptions onto TRIS beads (C<sub>0</sub>=100 mg/L, 298K, 48 h., 25mg TAPEHA /500ml, 0.1 M for Pd (II), 0.1 M for Au (III), respectively).

<b>Pd (II), time (min)</b>	<b>First experiment</b>		<b>Second experiment</b>		<b>Avarage q<sub>e</sub> (mg/g)</b>	<b>Standard deviation</b>
	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>		
60	85.2	296	92.6	148	222.0	104.7
120	88	240	86.2	276	258.0	25.5
240	78.5	430	81.5	370	400.0	42.4
360	73.6	528	71.6	568	548.0	28.3
480	70.3	594	68	640	617.0	32.5
600	70.5	590	66.5	670	630.0	56.6
1440	68.1	638	68.5	630	634.0	5.7
2880	67.5	650	68.5	630	640.0	14.1

<b>Au (III), time (min)</b>	<b>First experiment</b>		<b>Second experiment</b>		<b>Avarage q<sub>e</sub> (mg/g)</b>	<b>Standard deviation</b>
	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>	<b>C<sub>e</sub> (mg/L)</b>	<b>q<sub>e</sub> (mg/g)</b>		
60	93.1	138	92.5	150	144.0	8.5
120	91.1	178	88.9	222	200.0	31.1
240	86.2	276	85.8	284	280.0	5.7
360	80.1	398	79.9	402	400.0	2.8

480	73.2	536	74.8	504	520.0	22.6
600	72.7	546	71.3	574	560.0	19.8
1440	71.9	562	71.3	574	568.0	8.5
2880	71.9	562	71.1	578	570.0	11.3

**Table S7.** The effect of temperature datas on Pd (II) and Au (III) adsorption onto TRIS beads (24 hours, 25 mL, 10 mg TIRS, 0.1 M for Pd (II), 0.1 M for Au (III), respectively, two experimental replicates).

Pd (II), Temperature (K)	First experiment		Second experiment		Avarage $q_e$ (mg/g)	Standard deviation
	$C_e$ (mg/L)	$q_e$ (mg/g)	$C_e$ (mg/L)	$q_e$ (mg/g)		
298	29.1	177.25	31.9	170.25	173.8	4.9
308	31.1	172.25	32.3	169.25	170.8	2.1
318	32.2	169.5	31.2	172	170.8	1.8
298	34.1	164.75	33.5	166.25	165.5	1.1
Au (II), Temperature (K)	First experiment		Second experiment		Avarage $q_e$ (mg/g)	Standard deviation
	$C_e$ (mg/L)	$q_e$ (mg/g)	$C_e$ (mg/L)	$q_e$ (mg/g)		
298	34	165	36	160	162.5	3.5
308	31	172.5	29.4	176.5	174.5	2.8
318	22	195	23	192.5	193.8	1.8
328	18	205	19.2	202	203.5	2.1

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