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Effective recovery of $AuCl_4^-$ using thiosemicarbazide and thiocarbohydrazide functionalized D301 resin

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ABSTRACT

In this study, two novel adsorbents (TSCD301 and TCHD301) were successfully synthesized through ring-opening reaction between the amino of thiosemicarbazide or thiocarbohydrazide and the epoxy group of poly(glycidyl methacrylate). The adsorption performance and recognition selectivity of TCHD301 and TSCD301 toward $AuCl_4^-$ were studied by batch adsorption experiments. The results showed that the equilibrium adsorption capacity of TSCD301 and TCHD301 toward $AuCl_4^-$ could reach 410.76 and 566.59 $mg\cdot g^{-1}$ at 298 K and pH of 2, respectively. The adsorption process could be described using the pseudo-second-order model, and the adsorption behavior of $AuCl_4^-$ on TSCD301 and TCHD301 was typical monolayer adsorption. In mixed solution, two adsorbents had excellent selectivity toward $AuCl_4^-$. In addition, the adsorption capacity of adsorbent did not change significantly in repeated adsorption-desorption experiment.

Keywords: D301 resin; Modification; $AuCl_4^-$; Adsorption; Selectivity

1. Introduction

Gold is widely used in the fields of agriculture [1], medicine [2], catalyst [3,4], electronics [5,6] and sensor [7] due to its distinctive physical and chemical properties. In recent years, the demand of gold has been increasing in the industrial and economic fields, while the concentration of natural gold resource has been decreasing. Moreover, gold is a non-renewable resource. Consequently, it is particularly urgent to recover gold from secondary resources. Adsorption is an effective method to recover gold from the view of cost and operation [8,9].

Commonly used adsorbents are activated carbon [10,11], adsorption resin [12], cellulose [13] and silica gel [14,15]. Adsorption resin is not only cheap and available, but also has excellent mechanical strength and chemical stability. For adsorption resin, the adsorption ability depends on the activity and number of surface active sites [16]. So, it is an important and effective approach to improve the adsorption performance of existing traditional resins through chemical modification with special compounds.

In this study, two new adsorbents with abundant *S* and *N* donor atoms were successfully prepared through chemical modification. First, poly(glycidyl methacrylate) (PGMA) was grafted onto the surface of D301 resin. Then, TSCD301 and TCHD301 were obtained via ring-opening reaction between the amino and the epoxy group. The adsorption performance and recognition selectivity of two adsorbents toward $AuCl_4^-$ were investigated by batch adsorption experiments.

2. Experimental setup

2.1. Materials and instruments

D301 weak basic resin (Wandong Chemical Co., Ltd., Anhui, China). Glycidyl methacrylate (GMA, Xiayi Yuhao

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Additives Co., Ltd., Henan, China) was purified by distillation under vacuum. Ammonium persulfate (APS, Kaitong Chemical Reagent Co., Ltd., Tianjin, China). Chloroauric acid (Nanjing Chemical Reagent Co., Ltd., Nanjing, China). Thiosemicarbazide (TSC, Aladdin Biochemical Technology Co., Ltd., Shanghai, China). Thiocarbohydrazide (TCH, Baishun Chemical Technology Co., Ltd., Beijing, China). Thiourea and other reagents were purchased from Beijing Chemical Factory (Beijing, China). All reagents were analytical grade in this experiment.

FTIR-8400S infrared spectrometer (Shimadzu, Japan). ICP-AES spectrometer (Leeman Labs, America). PHS-3C acidity meter (Shanghai INESA Science Instrument Co., Ltd., China). THZ-82 type constant temperature shaker (Jintan Fuhua Instrument Co., Ltd., China).

2.2. Preparation and characterization of adsorbent

10 g of D301 resin and 200 mL of distilled water were mixed and stirred at 298 K for 8 h. The pretreated D301 resin was obtained after vacuum drying. 1 g of pretreated D301 resin and 10 mL of GMA were added into 100 mL of N,N-dimethylformamide (DMF). 0.1611 g of APS (initiator, 1.5 wt% of monomer) was added into the system after passing nitrogen for 30 min. Graft polymerization was performed under N_2 atmosphere at 313 K for 18 h. The product was extracted with ethanol and distilled water to remove the homopolymer on the resin surface. The graft material was obtained and labeled as D301-*g*-PGMA.

1.75 g (0.019 mol) of TSC or 1.5 g (0.014 mol) of TCH was dissolved into 50 mL of NaOH solution (pH = 14), then 0.1 g of D301-*g*-PGMA was added to the system and the ring-opening reaction was carried out at 353 K for 12 h. The products were constantly washed with distilled water until the filtrate was neutral. Finally, thiosemicarbazide and thiocarbohydrazide modified resins were obtained and labeled as TSCD301 and TCHD301, respectively. The preparation process of TSCD301 and TCHD301 is shown in Fig. 1.

In this study, the grafting degree of PGMA was 0.32 ± 0.02 $g \cdot g^{-1}$, and the ring-opening rate of both TCH and TSC was 55% ± 2%. The infrared spectra of the each step products were measured by KBr tablet method.

2.3. Batch adsorption experiments

0.01 g of TSCD301 or TCHD301 was mixed with 350 mL of $HAuCl_4$ solution and shaken in a constant temperature shaker. The influences of adsorption time, concentration, temperature and pH of the solution on the adsorption capacity were investigated. The concentration of $AuCl_4^-$ was determined using ICP-AES and the adsorption capacity (Q, $mg \cdot g^{-1}$) was calculated according to Eq. (1):

$$Q = \frac{V(C_0 - C_t)}{m} \tag{1}$$

where V (L) is the volume of $HAuCl_4$ solution, C_0 and C_t ($mg \cdot L^{-1}$) are the initial and residual concentration of $AuCl_4^-$ solution at t (h) time, m (g) is the mass of the adsorbents (TSCD301 and TCHD301).

2.4. Adsorption efficiency

In order to further evaluate the practical application ability of adsorbent, the influence of adsorbent dosage on adsorption efficiency was studied by batch adsorption experiments. Under the same conditions, the adsorbents of different mass were shaken together with 100 mL $HAuCl_4$ solution (30 $mg \cdot L^{-1}$), respectively. The adsorption efficiency (A_f) was calculated according to Eq. (2)

$$A_f(\%) = \frac{C_0 - C_t}{C_0} \times 100 \tag{2}$$

2.5. Adsorption selectivity

Usually, there are some coexisting ions in gold-bearing chloride solution, which will have a negative impact on the recovery of gold. In this study, the recognition ability of TSCD301 and TCHD301 toward $AuCl_4^-$ were investigated by batch method. In mixed solution, the concentrations of $AuCl_4^-$ and other metal ions (M) were 25 and 100 $mg \cdot g^{-1}$, respectively. The distribution coefficients (K_d) were obtained according to Eq. (3) and the selectivity coefficients (k) were calculated according to Eq. (4).

$$K_d = \frac{Q_e}{C_e} \tag{3}$$

$$k_{(AuCl_4^-/M)} = \frac{K_d(AuCl_4^-)}{K_d(M)} \tag{4}$$

where Q_e ($mg \cdot g^{-1}$) is the equilibrium adsorption capacity, C_e ($mg \cdot L^{-1}$) is the equilibrium concentration of $AuCl_4^-$ or other metal ions (M).

2.6. Repeated use experiment

In order to investigate the stability and reusability of the two adsorbents, the spent TCHD301 and TSCD301 were desorbed with thiourea (1 $g \cdot L^{-1}$). Then the adsorption-desorption cycle experiments were repeated 10 times.

3. Results and discussion

3.1. Characterization of adsorbent

The infrared spectra of D301, D301-*g*-PGMA, TSCD301 and TCHD301 are shown in Fig. 2.

It can be seen that the infrared spectrum of D301-*g*-PGMA shows the stretching vibration absorption peaks of ester carbonyl and epoxy group at 1,714 and 1,134 cm^{-1}, respectively. These indicate that PGMA has been successfully grafted onto the D301.

In the infrared spectra of TSCD301 and TCHD301, the new peaks at 1,363 and 1,357 cm^{-1} are assigned to the characteristic absorption peaks of C=S [17–19], the characteristic absorption peak of N–H at 3,450 cm^{-1} [20] is significantly enhanced, and the adsorption peak of epoxy group at 1,134 cm^{-1} is obviously weakened. These facts indicate that

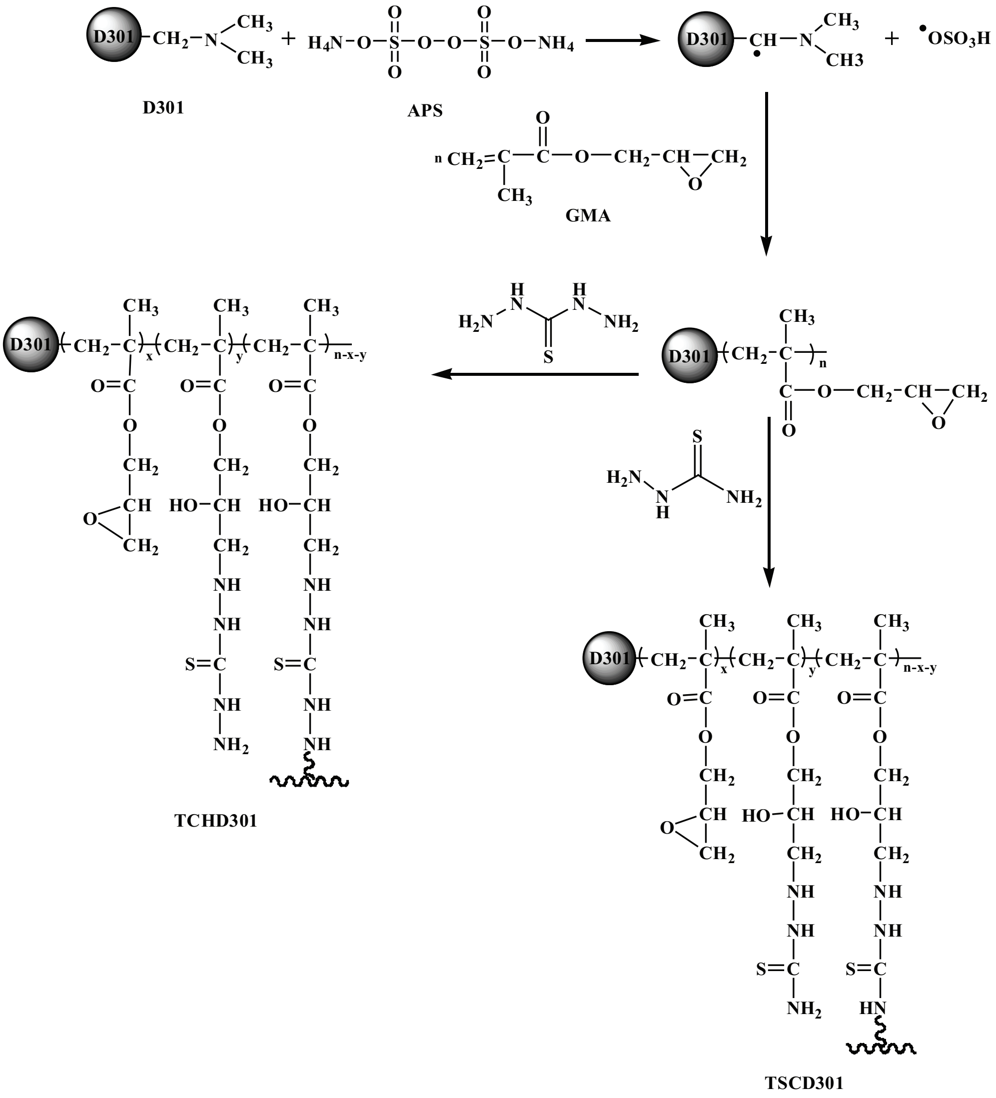

Fig. 1. Preparation process of TSCD301 and TCHD301.

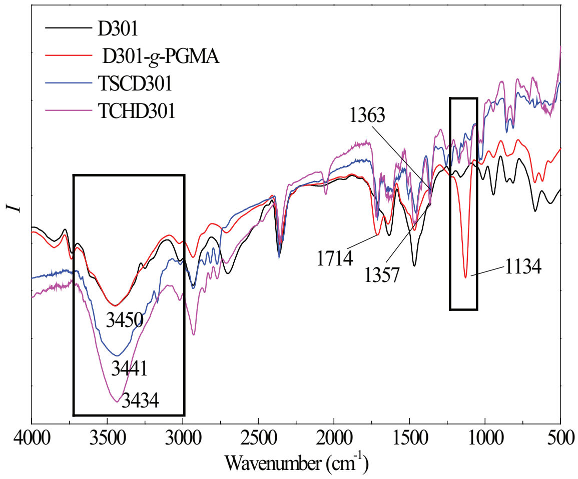

Fig. 2. FTIR spectra of D301, D301-*g*-PGMA, TSCD301 and TCHD301.

TSCD301 and TCHD301 resins are successfully obtained through ring-opening reaction.

Finally, the N–H absorption peak of TCHD301 (at 3,434 cm^{-1}) is stronger than that of TSCD301 (at 3,441 cm^{-1}), which suggests that the nitrogen content of TCHD301 is higher than that of TSCD301 under the same ring-opening rate.

3.2. Adsorption kinetics curves

The adsorption kinetics curves of TSCD301 and TCHD301 for $AuCl_4^-$ are shown in Fig. 3.

In Fig. 3, the equilibrium adsorption capacity of TSCD301 (at 20 h) and TCHD301 (at 18 h) toward $AuCl_4^-$ reached 410.76 and 566.59 mg·g^{-1}, respectively. In addition, the adsorption capacity of TSCD301, TCHD301 and other adsorbents toward $AuCl_4^-$ is listed in Table 1.

It can be seen that the adsorption capacity of TSCD301 and TCHD301 is higher than that of D301 and other adsorbents, which may be attributed to the properties of functional groups on the surface of adsorbents. On the one hand, in acidic media, the amino groups of TSCD301 and TCHD301 are protonated and transformed into positive charge centers. So, the $AuCl_4^-$ anions can be attracted onto the adsorbents by electrostatic interaction. On the other hand, according to the HSAB theory [36–38], there is strong coordination interaction between N or S donor atoms and $AuCl_4^-$.

In addition, TCHD301 has larger adsorption capacity and faster adsorption rate than TSCD301. This phenomenon can be explained by the number of active sites under the same ring-opening rate, which is accordance with nitrogen content of TCH and TSC molecules (TCH > TSC).

The experimental data in Fig. 3 are fitted by the Lagergren-first-order (Eq. (5)) and pseudo-second-order (Eq. (6)) kinetics models to further study the adsorption mechanism of TCHD301 and TSCD301 toward $AuCl_4^-$ The maximum adsorption capacity, adsorption rate constant and correlation coefficient (R^2) are obtained and listed in Table 2. In addition, the fitting curves are shown in Fig. 3.

$$\ln(Q_m - Q_t) = \ln Q_m - k_1 t \tag{5}$$

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_m^2} + \frac{t}{Q_m} \tag{6}$$

where Q_t ($mg\cdot g^{-1}$) is the adsorption capacity at t (h) time, Q_m ($mg\cdot g^{-1}$) is the maximum adsorption capacity, k_1 (h^{-1}) and k_2 ($g\cdot mg^{-1}\cdot h^{-1}$) are adsorption rate constants.

According to Table 2, the R^2 of pseudo-second-order model is higher than that of Lagergren-first-order model and its maximum adsorption capacity is closer to the experimental data. These facts combined with the fitting curves in

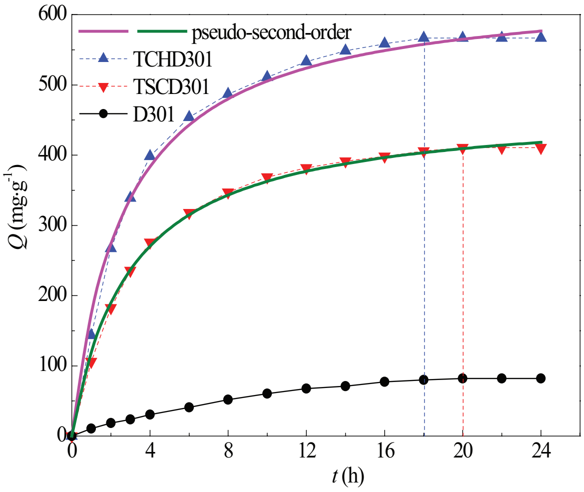

Fig. 3. Adsorption kinetics curves. $T = 298$ K, pH = 2.

Table 2
Fitting results of kinetics models

Adsorption model	Parameter	Adsorbent	
		TSCD301	TCHD301
Lagergren-first-order	Q_m	336.17	456.45
	k_1	0.21	0.22
	R^2	0.9959	0.9886
Pseudo-second-order	Q_m	469.48	641.03
	k_2	0.0007	0.0006
	R^2	0.9990	0.9981

Table 1
Adsorption capacity of different adsorbents toward $AuCl_4^-$

Adsorbent	Temperature (K)	pH	Q_{max} ($mg\cdot g^{-1}$)	Reference
Coconut shell carbon	298	11	1.79	[21]
MNP-G3	298	6.5	3.58	[22]
AC-ANPS	298	2	32.30	[23]
AC-TRIS	298	1	33.57	[24]
Taurine-modified cellulose	295	3.26	34.50	[13]
AC-dithizone	298	2	43.90	[25]
mGO@SiO_2@PPy-PTh	298	4.8	50.00	[26]
4-(aminomethyl) pyridine-Silica	298	4	55.50	[27]
SG–ClPrNTf_2	298	2	61.10	[28]
OPT-T	303	2	75.30	[29]
AS-5BA	298	1	87.75	[30]
PUF-Cyt	298	1	98.90	[31]
Cellulose acetate fibers	298	–	110.00	[32]
PPC-EG 0.5	298	2	157.61	[33]
APS-LCP	303	2	261.36	[34]
PS-APD resin	–	4	278.50	[35]
TSCD301	298	2	410.76	This study
TCHD301	298	2	566.59	This study

Fig. 3 confirm that the adsorption process of TSCD301 and TCHD301 toward $AuCl_4^-$ follows pseudo-second-order model. These further indicate that the concentration of $AuCl_4^-$ and the number of functional groups have a great influence on the adsorption performance [13].

3.3. Adsorption isotherms

The influences of $AuCl_4^-$ concentration (6, 12, 18, 24 and 30 mg·g⁻¹) on adsorption capacity were studied. The adsorption isotherms are shown in Fig. 4. In order to further describe the adsorption behavior of $AuCl_4^-$ on TSCD301 and TCHD301, Langmuir (Eq. (7)), Freundlich (Eq. (8)) and Sips (Eq. (9)) adsorption models are used to fit the experiment data of adsorption isotherms, and the fitting results are listed in Table 3.

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_0} + \frac{1}{K_L Q_0} \tag{7}$$

$$\ln Q_e = \ln k + \frac{1}{n}\ln C_e \tag{8}$$

$$Q_e = \frac{Q_0 b C_e^{\,m}}{1 + b C_e^{\,m}} \tag{9}$$

where Q_0 (mg·g⁻¹) is the monolayer adsorption capacity, K_L (L·mg⁻¹) is the Langmuir constant, k and n are Freundlich constants related to adsorption performance, b (L·mg⁻¹) is the adsorption equilibrium constant and m is the dissociation parameter. If b is close to 0, the Sips model will be transformed into Freundlich model; if m is close to 1, the Sips model will be transformed into Langmuir model.

In Table 3, compared with Langmuir model, the R^2 of Freundlich model is closer to 1 and the b of Sips model is closer to 0. These facts indicate that the adsorption fit well with Freundlich model and the adsorption behavior of $AuCl_4^-$ on adsorbent is a typical monolayer adsorption.

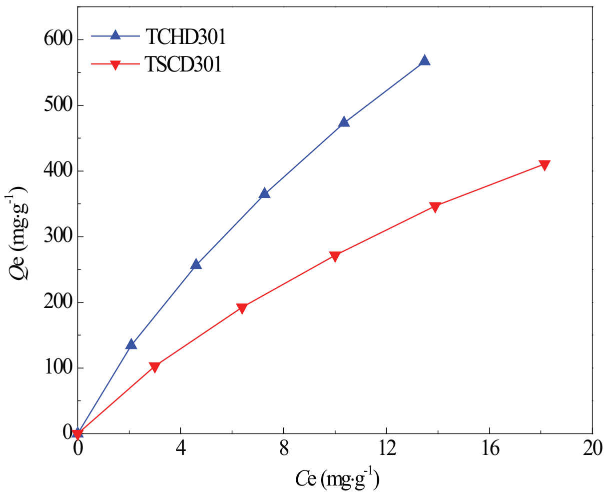

Fig. 4. Adsorption isotherms of TSCD301 and TCHD301 toward $AuCl_4^-$. T = 298 K, pH = 2.

Table 3
Fitting results of different adsorption models

Model	Parameter	Adsorbent	
		TSCD301	TCHD301
Langmuir	Q_0	1022.10	1390.56
	K_L	0.04	0.05
	R^2	0.9949	0.9872
Freundlich	n	1.29	1.30
	k	44.97	77.65
	R^2	0.9977	0.9987
Sips	Q_0	1266.70	1838.57
	b	0.0313	0.0393
	m	0.9432	0.9319
	R^2	0.9998	0.9999

3.4. The influence of temperature on adsorption capacity

The influence of temperature on adsorption capacity was investigated from 298 to 318 K. The results are shown in Fig. 5.

It can be seen that temperature has a significant influence on the adsorption capacity, and the adsorption capacity increases with the increase of temperature. The reason is that the molecular chains on the adsorbent surface can be stretched better at higher temperature, which improves the utilization rate of active sites.

3.5. The influence of pH on adsorption capacity

The influence of pH on adsorption capacity was studied in the range of 1–3. Because pH affects the surface speciation of adsorbent and the presence form of chlorogold compounds ($AuCl_4^-$) in aqueous solution [21,39,40]. The results are shown in Fig. 6.

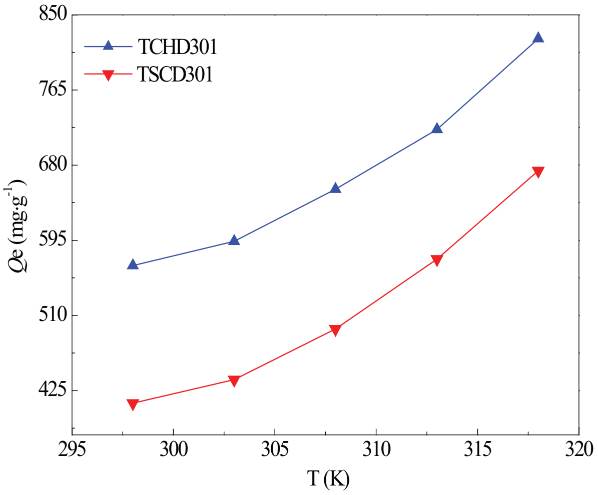

Fig. 5. Influence of temperature on adsorption capacity. pH = 2.

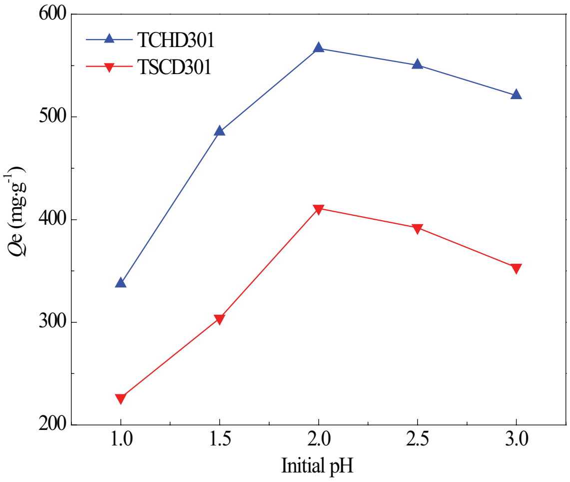

Fig. 6. Influence of pH on adsorption capacity. *T* = 298 K.

It can be seen that the pH has an important influence on the adsorption capacity of TSCD301 and TCHD301, and the adsorption capacity is the highest at the pH of 2.

When the pH value is greater than 2, the protonation degree of amino group decreases significantly, this weakens the electrostatic interaction and directly leads to the decrease of adsorption capacity. When the pH value is less than 2, the solution contains a larger amount of Cl^- (using HCl to adjust the pH of the solution), which is more likely to form electrostatic interaction with protonated amino groups. In addition, the protonation degree of amino group is significantly increased in the strongly acidic solution, resulting that the coordination interaction is hard to form. Consequently, when the pH value is 2, the adsorption capacity is the highest.

The possible adsorption path of TSCD301 and TCHD301 toward $AuCl_4^-$ are shown in Fig. 7. The interaction between TCHD301 or TSCD301 and $AuCl_4^-$ includes electrostatic interaction and coordination interaction [41–43].

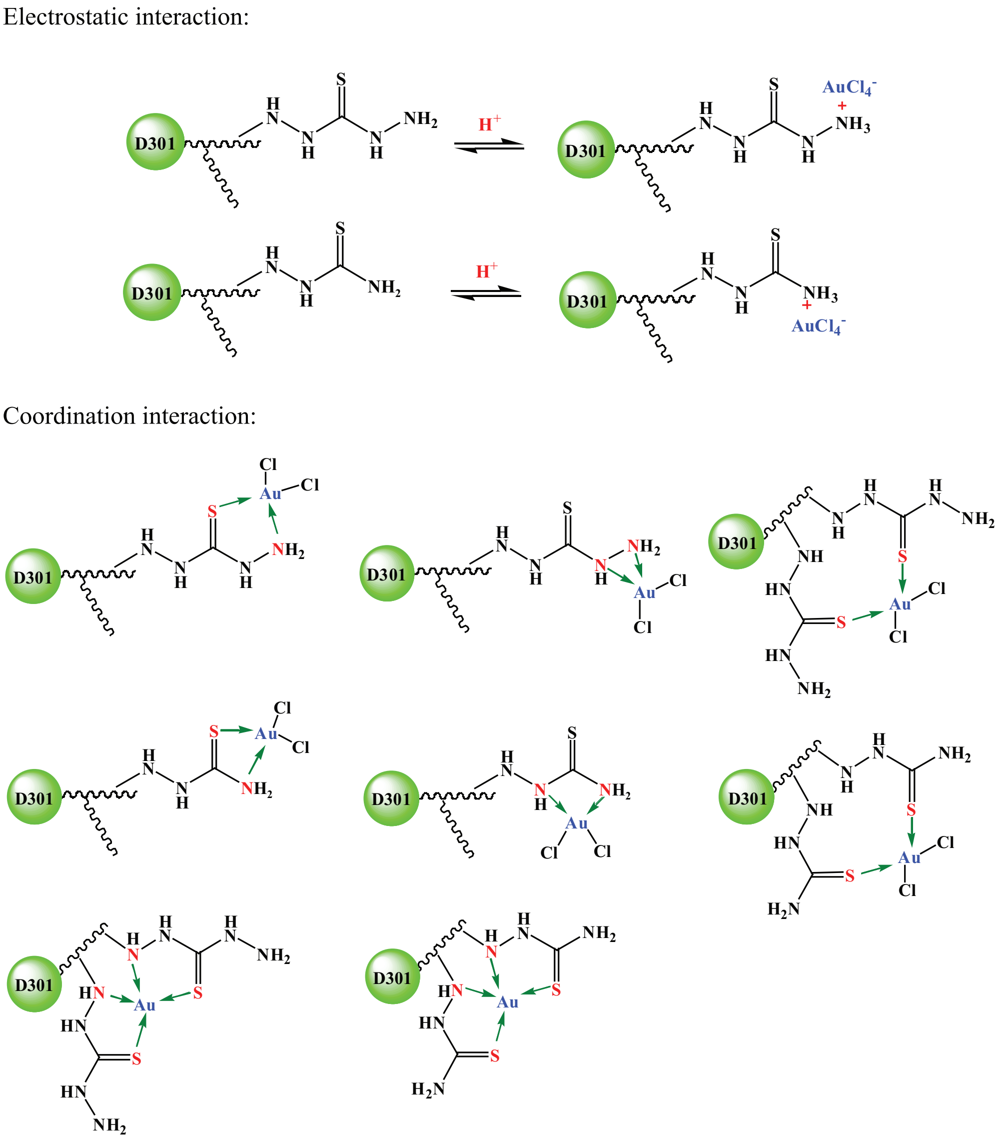

Fig. 7. Adsorption path of TSCD301 and TCHD301 toward $AuCl_4^-$.

XPS spectrum of TSCD301 after adsorbing AuCl$_4^-$ is shown in Fig. 8.

In Fig. 8, the adsorption peaks of Au$_4$f and Cl$_2$p can be clearly observed. These indicate that AuCl$_4^-$ has been adsorbed onto the surface of TSCD301.

XPS spectrum of Au$_4$f is shown in Fig. 9.

In Fig. 9, new absorption peaks of Au$_4$f appear at 86.98 and 83.27 eV, which is lower than the original absorption peaks of Au$_4$f (87.66 Au$_4$f$_{5/2}$, 84.01 Au$_4$f$_{7/2}$). In addition, the absorption peak of Au^0 was not observed in Fig. 9, indicating that Au did not undergo a reduction reaction during the adsorption process.

3.6. The influence of adsorbent dosage on adsorption efficiency

The influences of adsorbent dosage (0.01, 0.02, 0.03, 0.04 and 0.05 g) on adsorption efficiency are shown in Fig. 10.

When the adsorbent mass is 0.05 g, the adsorption efficiency of TSCD301 and TCHD301 reached 98.23% at 1.5 h and 1 h, respectively. Besides, all adsorption efficiency can reach a certain value under different mass conditions, which follows a rule that the more the adsorbent dosage, the faster the adsorption rate. The reason is that the number of active sites increases with the adsorbent mass. At the same time, these facts prove that TSCD301 and TCHD301 can be used to effectively recover AuCl$_4^-$ from the low concentration of secondary resources.

3.7. Adsorption selectivity

In mixed solution, the recognition selectivity of TSCD301 and TCHD301 toward AuCl$_4^-$ was investigated. The distribution coefficients and selectivity coefficients are listed in Table 4.

It can be seen that the selectivity coefficients of TSCD301 and TCHD301 are higher than that of D301. This indicates that TSCD301 and TCHD301 have excellent selectivity toward AuCl$_4^-$. Therefore, these two adsorbents can be used to selectively adsorb AuCl$_4^-$ from gold-bearing chloride solution.

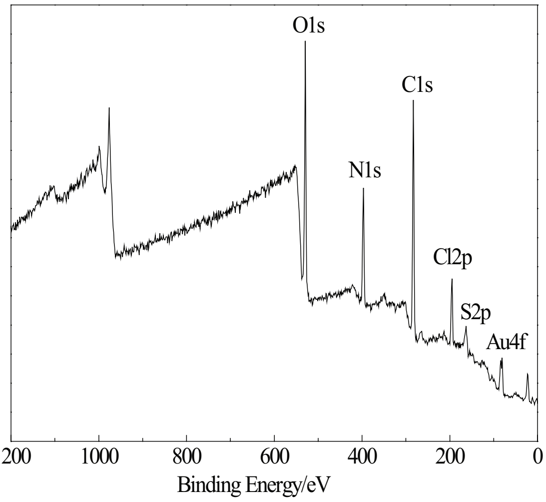

Fig. 8. XPS spectrum of TSCD301 after adsorbing AuCl$_4^-$.

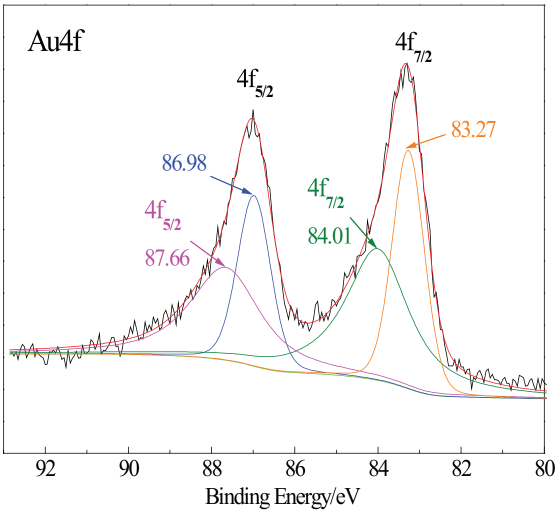

Fig. 9. XPS spectrum of Au$_4$f.

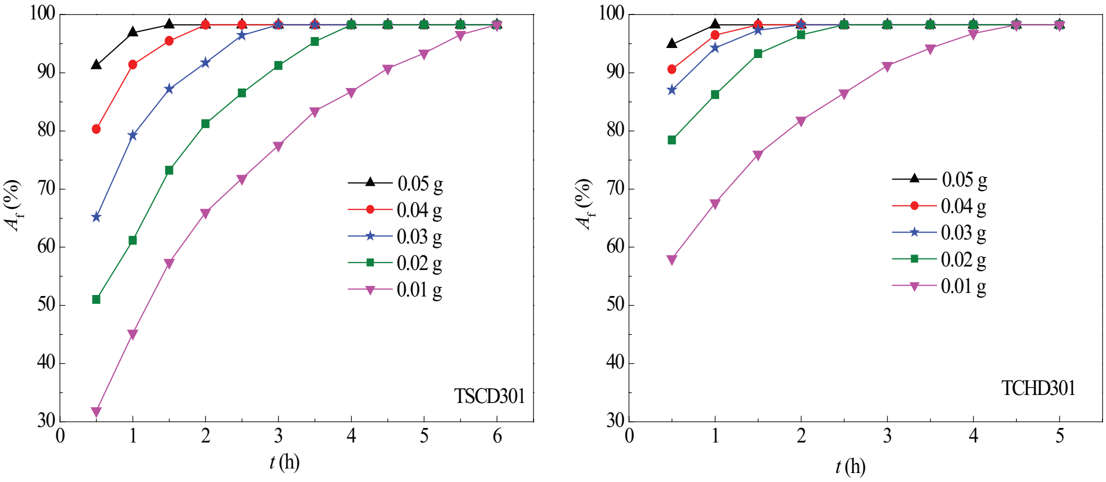

Fig. 10. The influence of adsorbent dosage on adsorption efficiency. *T* = 298 K, pH = 2.

Table 4
Distribution coefficient and selectivity coefficient data

Adsorbent	Coefficient	Mixture system							
		$AuCl_4^-$	$CuCl_4^{2-}$	$AuCl_4^-$	Zn^{2+}	$AuCl_4^-$	Fe^{3+}	$AuCl_4^-$	Ni^{2+}
D301	K_d ($L\cdot g^{-1}$)	2.6010	0.2301	3.1932	0.0561	2.6483	0.1758	3.3106	0.0114
	$k_{(AuCl_4^-/M)}$	11.30		56.92		15.06		290.40	
TSCD301	K_d ($L\cdot g^{-1}$)	28.2303	0.0700	32.8947	0.0463	31.5759	0.0638	33.0679	0.0355
	$k_{(AuCl_4^-/M)}$	403.29		710.47		494.92		931.49	
TCHD301	K_d ($L\cdot g^{-1}$)	55.6203	0.1000	72.5036	0.0974	56.0696	0.0998	81.5981	0.0859
	$k_{(AuCl_4^-/M)}$	556.20		744.39		561.82		949.92	

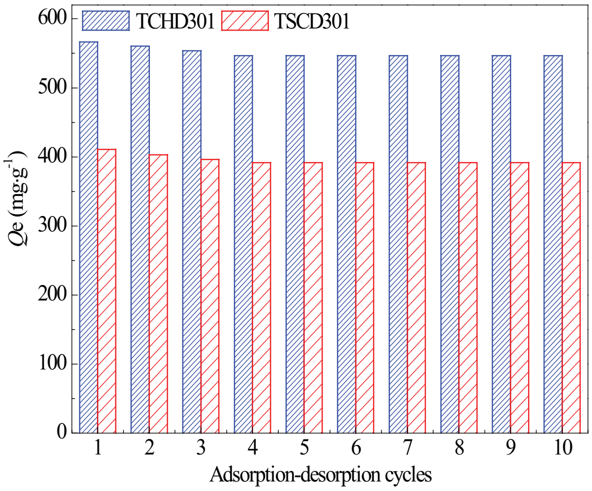

Fig. 11. Adsorption-desorption cycles of TSCD301 and TCHD301. T = 298 K, pH = 2.

3.8. Desorption and reusability

In order to test the stability and reusability of adsorbent, the adsorption-desorption experiments were repeated 10 times. The results are shown in Fig. 11.

It can be seen that the adsorption capacity of TSCD301 and TCHD301 did not change significantly during the reuse process. This fact indicates that thiourea (1 $g\cdot L^{-1}$) is an effective desorption solution. At the same time, this fact indicates that the chemical composition and structure of the adsorbents will not be destroyed during regeneration process.

4. Conclusions

In this study, TSCD301 and TCHD301 were successfully prepared through ring-opening reactions between the amino of thiosemicarbazide or thiocarbohydrazide and the epoxy group of PGMA. The results showed that adsorption time, pH, temperature and solution concentration had a significant influence on the adsorption performance. TSCD301 and TCHD301 have high adsorption capacity toward $AuCl_4^-$. The adsorption kinetics follows pseudo-second-order model and the adsorption behavior is a typical monolayer adsorption. In addition, TSCD301 and TCHD301 have good chemical stability and excellent recognition selectivity. TSCD301 and TCHD301 can be used as two promising adsorbents to effectively and selectively recover gold from aqueous solution systems.

Acknowledgement

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