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Study on the adsorption behavior of modified persimmon powder biosorbent on Pt(IV)

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Abstract A glycine-modified persimmon powder was used as biosorbent for platinum adsorption in aqueous solution. The combined effects of modifying agent amount, modifying time and temperature on Pt(IV) adsorption by persimmon biosorbent were investigated, from which the optimum modification conditions with modifying agent amount of 50 %, temperature of 333 K and contact time of 4.0 h were determined. The influences of solution initial pH, solid-to-liquid ratio, contact time, temperature and initial concentration on the Pt(IV) adsorption were studied systematically by batch experiments. The optimum modification conditions were determined as follows: chemical reagent amount of 50 %, temperature of 333 K and contact time of 4.0 h. From the adsorption kinetic analysis, it was found that the adsorption kinetics of glycine-modified persimmon powder followed a pseudo-second-order kinetic model. The adsorption isotherms of the crude persimmon powder and glycine-modified persimmon powder were analyzed with the BET-type adsorption isotherm. The adsorption mechanism of Pt(IV) on the glycine-modified persimmon was considered to be adsorption-reduction mechanism. The results achieved from this study indicated that the novel persimmon adsorbent could be applied to recover trace Pt(IV) ions from a concentrated chloride solution.

Keywords Persimmon · Glycine · Platinum · Biosorption · BET isotherm

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Introduction

Platinum is widely used in ornaments, catalytic agent, energy materials and so on. Due to its high chemical stability as well as high conductivity, its consumption is growing increasingly in the past years. Because of the increasing demand along with the scarcity of natural platinum resources, how to efficiently treat the lowgrade natural ores and secondary resources containing platinum group metals has attracted much attention. In general, aqua regia and hydrochloric acid containing chloride ions are utilized to separate and recover platinum group metals from natural or secondary platinum resource (Parajuli et al. 2008a). Conventional methods used for recovering platinum from this type of leaching liquor include ion exchange, electrowinning, solvent extraction, etc. Certainly, these processes have several drawbacks including lower recovery efficiency, secondary contamination and cost ineffectiveness. Recently, biosorption technology has been found to be a simple and clean method to recover platinum from aqueous solution. Some of the adsorbents such as orange peel, grape wastes and rice husk (Parajuli et al. 2006; Ramesh et al. 2008; Parajuli et al. 2009) were used for removing toxic elements and recovering precious metals from aqueous solution.

Waste persimmon generated from forestry and agriculture is composed of tannin, pectin, cellulose and other low molecular weight hydrocarbons (Parajuli et al. 2008a). Some researchers have reported that the crosslinking persimmon exhibited outstanding adsorption capacity for Au(III) and the maximum platinum adsorption capacity reached 45.8 mg/g when using the bayberry tannin immobilized collagen fiber membrane (Gurung et al. 2011; Ma et al. 2006).



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This paper studies a novel glycine-modified persimmon powder for Pt(IV) from aqueous solution, and the adsorption mechanism of GPP is addressed. The modification is to introduce glycine into persimmon powder (abbreviated as PP) to synthesize an effective adsorbent based on Mannich reaction shown below.

The effects of various parameters including solution initial pH, contact time, temperature and solid-to-liquid ratio (S/L) on the adsorption efficiency of Pt(IV) were investigated.

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Location of date of the research carried out is at School of Metallurgical Science and Engineering, Central South University, Changsha, China.

Materials and methods

Materials

Stock solution was prepared by diluting analytical-grade $H_2PtCl_6 \cdot H_2O$ using double-distilled water. HCl and NaOH with analytical purity were used to control the solution pH. The analytical-grade $H_2PtCl_6 \cdot H_2O$ was provided by National Medicine Group Chemical Reagent Co., LTD. The crude waste persimmon used was kindly supplied by Bio Technology Co., in Shanxi Province of China.



Fig. 1 Effect of chemical reagent amount, temperature and reaction time on persimmon modification. **a** The effect of chemical reagent amount (Conditions: temperature—333 K; reaction time—4 h; force air drying); **b** the effect of temperature (Conditions: chemical reagent

amount—50 %; reaction time—4 h; force air drying); **c** the effect of reaction time. (Conditions: chemical reagent amount—50 %; temperature—333 K; force air drying)



Preparation of persimmon powder (PP)

The waste persimmon consisted of astringent persimmon fruit, was washed with double-distilled water and dried at 333 K in a convection oven for 24 h. The dried persimmon was crushed and sieved to make the particle size lower than 0.40 mm. Then, the obtained product was named as PP for further adsorption study.

Modification experiments

Modification experiments of PP were carried out by taking certain amounts of PP and modified agent (a mixture solution composed of glycine, formaldehyde and concentrated hydrochloric acid) together in a shaking bath by controlling definite modified temperature and contact time. Then, the modified product was filtered and washed with double-distilled water and dried in convection oven for 24 h. Finally, through grinding and sieving, the glycine-modified persimmon powder (hereafter abbreviated as GPP) was obtained.

Adsorption experiments

A series of batch adsorption experiments for Pt(IV) were individually conducted in the shake bath under certain conditions by using GPP and PP as adsorbent, respectively. The effects of various parameters, including solution initial pH, solid-to-liquid ratio, temperature, and adsorption kinetics and adsorption isotherms on the Pt(IV) adsorption process, were investigated. The adsorption kinetics of Pt(IV) were studied by taking 10 mL of Pt(IV) solution (30 mg/L) together with 50 mg of GPP and shaking the mixture at various temperatures (303, 328 and 343 K). Adsorption efficiency of Pt(IV) and the amount of adsorption (q, mmol/g) were calculated according to Eqs. 1 and 2, respectively:

Adsorption
$$\% = \frac{C_0 - C_e}{C_0} \times 100\%$$
 (1)

$$q = \frac{(C_0 - C_e)V}{m} \tag{2}$$

where C_0 and C_e represent the initial and equilibrium Pt(IV) concentration (mmol/L), respectively; *V* represents the volume of the solution (L), and *m* is the mass of adsorbent (mg).

Characterization methods

The main functional groups on PP and GPP were characterized with a Fourier transform infrared spectrophotometer (Nicolet 380). Metal concentrations were measured by using a WFX-130-B atomic absorption spectrophotometer (AAS). The oxidation state of the platinum bound the biomaterial GPP was ascertained with X-ray photoelectron spectroscopy (K-Alpha 1063).

Results and discussion

Chemical modification of persimmon powder (PP)

Effect of modifying agent amount

In this paper, the modifying agent was a mixture solution consisting of glycine, formaldehyde and concentrated hydrochloric acid. The amount of modifier changed within the range of 10-60 % (w/w% calculated based on the mass of PP); the efficiency of modified GPP was addressed by adsorption of Pt(IV), and the results are presented in Fig. 1a. It shows that the adsorption ability of GPP on Pt(IV) can be enhanced when increasing the modifier amount. This is because that modification reaction of PP is promoted and almost all of PP has been converted to GPP as the modifying reagent content increasing.

Effect of modification temperature

In order to elucidate the effect of temperature on the modification of PP, the experiments were conducted with the mixtures containing 1.0 g PP and 0.5 g modification reagent at various temperatures to address the behavior of GPP on adsorption of Pt(IV) and the results are shown in Fig. 1b. It can be seen that when the temperature increases from 293 to 333 K, the Pt(IV) adsorption by GPP increases slowly. Further increasing modified temperature, the Pt(IV) adsorption tends to decrease, which can be ascribed to the dissolution and decomposition of active ingredients in persimmon, thus hindering the Mannich reaction occurrence (Adhikari et al. 2008).

Effect of modification time

In these experiments, the modification time is optimized in the range of 0-12 h. The results shown in Fig. 1c illustrate that during the first four hours of reaction, the Pt(IV) adsorption by GPP is enhanced (Ren et al. 2007). Then, the Pt(IV) adsorption tends to be constant along with further increasing of time.

In conclusion, the optimum modification conditions were determined as follows: chemical reagent amount of 50 %, temperature of 333 K and temperature of 4.0 h.

FT-IR analysis

The FT-IR spectrums of PP and GPP obtained under the optimum modification conditions are shown in Fig. 2. For PP, the broad adsorption peak at 3288.0 cm^{-1} corresponds to the O–H stretching vibrations of tannin, pectin and cellulose, thus showing the presence of "free" hydroxyl groups on the adsorbent surface. The peak observed at





Fig. 2 FT-IR spectrums of PP and GPP

2936.2 cm⁻¹ can be attributed to the C-H stretching vibrations of methyl and methylene (Feng et al. 2009). The peaks observed at 1724.4 cm⁻¹ can be attributed to the carbonyl (C=O) stretching vibration of free carboxyl groups of catechin, catechol and gallic acid (Kim et al. 2007). The peaks at 1604.6 and 1458.2 cm^{-1} are due to the carbonyl (C=O) symmetrical and asymmetrical stretching vibrations of free ionization carboxyl in pectin and tannin acid. The vibration of hydroxyl (-OH) of aryl group in tannin and lignin appears at 1375.8 and 1205.7 cm^{-1} . The peaks at 932.0 and 877.1 cm^{-1} show the C=C bond stretching vibration. The broad peak at 3288.0 cm^{-1} in the PP becomes narrow and shifts to 3409 cm^{-1} in the GPP, which indicates that the O-H is involved in the modification. The shifting of the free ionization carboxy (C=O) peaks is attributed to the polymerization during the modification (Liang et al. 2010a). Moreover, two new peaks at 1315.7 and 459.1 cm^{-1} are due to amino (N-H) stretching, which infers the polyphenol groups have combined with glycine (Xiong et al. 2009).

Adsorption experiments

Effect of initial solution pH on Pt(IV) adsorption

The solution pH can influence the functional groups on the bio-absorbents and ion behavior in aqueous solution. Figure 3a gives the effect of initial solution pH on adsorption of Pt(IV) by PP and GPP. It is found that GPP shows better adsorption ability than that of PP. The adsorption efficiency increases as the initial solution pH changes from 0.4 to 2.4. And when the initial solution pH is above 2.4, the adsorption efficiency decreases slightly. When the pH value is below 0.4, the concentration of PtCl₆⁻ increases in the solution, which hinders the adsorbing of Pt(IV) (Gurung et al. 2011). Through



increasing solution initial pH properly, the activity of $PtCl_6^-$ can be enhanced and more Pt(IV) will be bound on the adsorbents, thus leading to the increase in the adsorption efficiency. Further increasing of pH may lead to hydrolysis of $PtCl_6^-$ to form Pt (OH)₂, and $Pt(OH)_4$ (Byrne and Yao 2000; Parajuli et al. 2009).

Effect of temperature on Pt(IV) adsorption

The effect of temperature on the adsorption efficiency of Pt(IV) is studied over the temperature range of 298–358 K. The results are presented in Fig. 3b. It is shown that the Pt(IV) adsorption on GPP is strengthened by increasing temperature below 343 K. Further increasing the temperature has less effect on the adsorption efficiency. This is because that high temperature enhances adsorption, (Feng et al. 2009; Cox et al. 2005). However, temperature has a negative effect on the adsorption when using PP, which may be attributed to increased solubility of PP which consequently weakens the Pt(IV) adsorption by PP as increasing the temperature (Duan et al. 2007).

Effect of solid-to-liquid ratio(S/L) on Pt(IV) adsorption

In order to determine an efficient and cost-effective process to recover platinum, the optimum solid-to-liquid ratio (mass of adsorbent to volume of metal ion solution, S/L) was determined as shown in Fig. 3c. It is illustrated that the adsorption efficiency of GPP is enhanced as S/L increases from 1.0 to 11.0 g/L. Further increasing S/L has a negligible impact on the adsorption efficiency. It is supposed that most of active metal ions can been adsorbed on the surface of GPP when the S/L reaches 11.0 g/L. However, PP shows a reverse tendency compared to GPP, which may be mainly attributed to the aggregation of PP in the solution hindering the adsorption of Pt(IV). When S/L is lower than 7.0 g/L, the aggregation of PP plays a primary role in the adsorption process, thus leading to the decrease in adsorption efficiency. When S/L is extremely large, despite of the presence of aggregation, some spare PP still can be remained to adsorb Pt(IV).

Adsorption kinetics of Pt(IV)

The investigation of contact time effect was carried out over the time range of 30–720 min at different temperatures, and the results are presented in Fig. 4. It can be seen that adsorption equilibrium at 298, 328 and 343 K could be reached within 8, 6 and 4 h, respectively.

The adsorption kinetics of Pt(IV) on GPP is studied by monitoring the Pt(IV) concentration remained in the aqueous solution at different temperatures. The observed data are analyzed in terms of the pseudo-second-order model which assumes that the efficiency is proportional to the square of the number R. The kinetic model can be expressed as Eq. 3 (Liang et al. 2010b):

$$\frac{t}{q_{\rm t}} = \frac{1}{kq_{\rm e}^2} + \frac{t}{q_{\rm e}} \tag{3}$$

where q_e and q_t are the amounts of the adsorption of Pt (mmol/g) at equilibrium and at time $t(\min)$, respectively; k is the constant of pseudo-second-order rate. The equilibrium adsorption amount q_e and pseudo-second-order efficiency constant k can be derived from the slope and the intercept of the plot t/q_t . The results are shown in Table 1. From Table 1, $R^2 > 0.9998$ is obtained for different temperature and theoretical values of q_e can agree well with the experimental values, indicating that the adsorption is fit

Moreover, according to the Eqs. 4 and 5, the standard free energy (ΔG°), enthalpy (ΔH°) and entropy change (ΔS°) thermodynamic parameters were calculated and illustrated in Table 2.

$$K_{\rm c} = C_{\delta}/C_{\rm e} \tag{4}$$

$$\ln K_{\rm c} = -\frac{\Delta G^{\rm o}}{\rm RT} = -\frac{\Delta H^{\rm o}}{\rm RT} + \frac{\Delta S^{\rm o}}{\rm R}$$
(5)

where K_c is the thermodynamic equilibrium constant, C_{δ} is mg of adsorbate adsorbed per liter and C_e is the equilibrium concentration of solution, mg/L.



Fig. 3 Effect of solution initial pH, temperature and S/L on the adsorption efficiency of Pt(IV). **a** The effect of solution initial pH (Conditions: temperature—333 K; S/L: 5.0 g/L; time: 24 h; Pt(IV) concentration—0.15 mmol/L); **b** the effect of temperature

(Conditions: initial pH: 0.9; S/L—5.0 g/L; time—24 h; Pt(IV) concentration—0.15 mmol/L); c the effect of S/L (Conditions: Pt(IV) concentration—0.15 mmol/L; solution pH—0.9; temperature—343 K; time—24 h)



From Table 2, it can be seen that the standard free energy (ΔG°) is negative at 298, 328 and 343 K, which indicates the adsorption process can occur spontaneously, while the values of ΔH° and ΔS° calculated from the slope and the intercept of the Eq. 4 are 143.90 and 0.53 kJ/mol, respectively. According to the study of Liu et al. (Liu and Liu 2008b), the enthalpy change (ΔH°) of physical adsorption is below 4.18 kJ/mol while that of chemical adsorption is over 20.92 kJ/mol. Based on the values of enthalpy change ΔH° , it is concluded that the Pt(IV) adsorption on the GPP studied here would be attributed to a chemical adsorption process. The positive value of ΔH° indicates the endothermic nature of process by GPP. The low value of ΔS° may suggest that no considerable change in entropy occurs during the Pt(IV) adsorption process on the GPP.



Fig. 4 Effect of contact time on the adsorption efficiency of Pt(IV). (Conditions: solution pH: 0.9; S/L: 5.0 g/L; Pt(IV) concentration: 0.15 mmol/L)

 $\label{eq:table_$

Temperature	$q_{\rm t} \; ({\rm mmol/g})$	Pseudo-second-order kinetic model			
(K)		$q_{\rm e} \ ({\rm mg/g})$	<i>K</i> [g (mg min)]	R^2	
298	0.12	0.12	0.0025	0.9999	
328	0.17	0.17	0.0028	0.9998	
343	0.17	0.17	0.0027	0.9998	

 Table 2
 Thermodynamic parameters for the adsorption of Pt(IV) by
 GPP

Adsorbent	Temperature (K)	K _c	$\Delta G^{\rm o}$ (kJ/mol)	$\Delta H^{\rm o}$ (kJ/mol)	$\Delta S^{\rm o}$ (kJ/mol)
GPP	298	1.91	-16.67	143.90	0.53
	328	2.51	-33.54		
	343	2.65	-40.51		

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Fig. 5 Effect of initial Pt(IV) concentration on the adsorption efficiency of Pt(IV). (Conditions: solution pH: 0.9; S/L: 5.0 g/L; time: 24 h; temperature: 343 K)

Adsorption isotherms of Pt(IV)

Figure 5 shows the effects of initial Pt(IV) concentration on the adsorption by PP and GPP. As revealed, Pt(IV) adsorption capacities of both adsorbents tend to approach the constant values at around 100 mg/g. Interestingly, Pt(IV) adsorption capacity of GPP continues to rise with a further increasing in Pt(IV) concentration, which is consistent with a typical BET-type adsorption isotherm based on multilayer adsorption model. Such BET type of adsorption behavior for Pt(IV) is also reported on lemon peel gel (Gurung et al. 2011). Consequently, the platinum adsorption capacity of GPP reaches as high as 250 mg/g.

Additionally, the results also indicate that the glycinemodified persimmon (GPP) is more effective than the crude persimmon powder (PP) for Pt(IV) adsorption from hydrochloric acid. It may be due to the improvement in surface morphology of the polymer matrices after chemical modification, which makes the GPP be mechanically strong and practically insoluble under the experimental condition. This is benefit for adsorption Pt(IV) from a concentrated chloride solution (Nakajima et al. 2003).

Table 3 shows the comparison of the maximum Pt(IV) adsorption capacities of different biomass-based adsorbents reported in different literatures. It can be seen the adsorption capacity of Pt(IV) by GPP in this work demonstrates remarkably highest capacity than that of other adsorbents.

Adsorption mechanism

Such a considerably high loading capacity of tannin-based adsorbents is supposed to involve the reduction in adsorbed Pt(IV) to the metallic Pt by polyphenols in the tannin molecules in persimmon.

 Table 3
 Adsorption capacities
 of different adsorbents toward Pt(IV)

Adsorbents	$q \pmod{g}$	Conditions	References
DMA persimmon waste gel (DMA-PW)	0.28	HCl 0.1 mol/L	Xiong et al. (2009)
Crosslinked grape waste	0.29	pH = 1.0	Parajuli et al. (2008a)
Crosslinked lignophenol gel	0.56	HCl 0.5 mol/L	Parajuli et al. (2008b)
Collagen fiber immobilized bayberry tannin	0.61	HCl 0.1 mol/L	Ma et al. (2006)
Carbonaceous sorbent prepared from flax shive	0.98	pH = 1.0	Cox et al. (2005)
GPP	1.28	pH = 1.0	This work
PP	0.69	pH = 1.0	This work



Fig. 6 Mechanism analysis of platinum adsorption on GPP and PP. a The EDS image of GPP after adsorbed platinum; b inferred mechanism of platinum adsorption; c high-resolution XPS spectra of the Pt-loaded GPP

Through introducing the electron-donating group (NH₂-CH₂COOH) to the aromatic rings, the interaction between Pt(IV) and the polyphonic groups in the tannin at acidic pH will be enhanced. However, this interaction is mostly dependent on the species of Pt(IV) existed in hydrochloric acid solution. As reported in the literatures, the chemical form of Pt(IV) is mainly $PtCl_6^{2-}$ in aqueous solution at pH of 1.0 (Chu 1983). Figure 6a shows the EDS of GPP-loaded platinum. As seen, the main elements of the adsorbent were C, Cl and Pt, which demonstrates that platinum in the aqueous solution can be adsorbed on the GPP through chelating, ionic-exchange and reduction.

Moreover, the standard reduction potentials of $E^{\Theta}_{\mathrm{Pt}^{4+}/\mathrm{Pt}^{2+}}$ and $E^{\Theta}_{\mathrm{Pt}^{2+}/\mathrm{Pt}^{0}}$ are 0.68 and 0.73 V, respectively, which are significantly higher than that of H⁺ (the standard reduction potential is 0.0 V), thus ensuring the reduction reaction of Pt(IV) preferentially ongoing (Chu 1983) as the reaction of tannin with Au(IV) (Gurung et al. 2011). Therefore, the mechanism of platinum adsorption on the GPP and PP consists of three steps as presented in Fig. 6b:

- The electrostatic adsorption step: the anionic species of 1. platinum combines with the positively charged adsorbent GPP.
- The reduction step: high valance of platinum is 2. reduced to low valance platinum Pt(II).
- 3. The aggregation step: the elemental platinum aggregates to the bigger particles of platinum.



To test the mechanism of Pt(IV) adsorption, it is very important to ascertain the oxidation state of the platinum bound on the biomaterial. Thus, high-resolution XPS spectra was carried out, and the data are collected from the Pt 4f core regions of the Pt-loaded biomaterial as well as standard Pt(IV) and Pt(IV) compounds (Fig. 6c). As shown in this figure, the spectra of the Pt-loaded biomaterial are well matched with those of standard Pt(IV) compounds (K₂PtCl₄). This result states that the platinum bound on the biomaterial is mostly or totally in divalent form. Therefore, it can be concluded that the adsorption mechanism of Pt(IV) on the GPP is the adsorption-reduction mechanism, that is, Pt(IV) can be reduced to Pt(IV) in the aqueous and solid phase.

Conclusion

The glycine-modified persimmon adsorbent (GPP) studied in this paper has considerable potential as an efficient and useful adsorbent to recover Pt(IV) from a hydrochloric acid solution. FITR study illustrated the involvement of amino groups in the Mannich reaction process. The adsorption efficiency of Pt(IV) of GPP reached 96 % under the optimum modification conditions with modified agent amount of 50 %, modified time of 4 h and temperature of 353 K. For PP and GPP, solution initial pH had an obvious influence on adsorption efficiency. When pH value was 2.4, almost 93 % Pt(IV) ions were adsorbed. All of active platinum ions had been adsorbed on the surface of GPP when the S/L was 11.0 g/L. The Pt(IV) adsorption kinetics followed the pseudo-second-order kinetics. A typical BET-type adsorption isotherm was applied to explain the Pt(IV) adsorption based on multilayer adsorption model. The high platinum uptake capacity of GPP of 250 mg/g was attributed to the adsorption-coupled reduction. From these results, it is concluded the glycine-modified persimmon (GPP) can be expected to be used to recover platinum group metals from a concentrated hydrochloric acid solution because of its chemical stability high adsorption capacity and fast efficiency.

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