Preconcentration and Determination of Au(III), Pd(II), and Pt(IV) Using Dispersive Micro-solid Phase Extraction with Multi-porous $ZnFe_2O_4$ Nanotubes and ICP-MS

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ABSTRACT

In this work, magnetic $ZnFe_2O_4$ nanotubes (ZFONTs) were used as a dispersive micro-solid phase extraction adsorbent for the separation and preconcentration of Au(III), Pd(II), and Pt(IV) and determination by inductively coupled plasma mass spectrometry. The experimental results showed that Au(III), Pd(II), and Pt(IV) can be adsorbed quantitatively on the ZFONTs in the pH range of 1.0–5.0, and then eluted completely with 1.0 mL of 1.5% thiourea in 0.5 mol L^{-1} HNO_3 solution. No carryover was observed in the next analysis.

Separation of the sorbent from the aqueous phase was carried out by an external magnet to avoid the time-consuming column passing or filtration/centrifugation steps. The parameters influencing the preconcentration and determination of the analytes were examined in detail. Under the optimized conditions, the detection limits of this method were 0.35, 0.17, and 0.64 pg mL^{-1} for Au, Pd, and Pt, respectively. Precision expressed as the relative standard deviation was less than 6.0% (n= 9, c=1.0 ng mL^{-1}). The developed method was applied to the determination of trace/ultra-trace Au, Pd, and Pt in biological and environmental certified reference materials, and the determined values were in good agreement with the certified values.

INTRODUCTION

Gold (Au), palladium (Pd), and platinum (Pt) are known as precious metals, and are widely used in different areas, including the petroleum, chemical, metallurgical, electrical, aerospace, nuclear power, and medical industries. As a result of their extensive application, they are inevitably discharged into the environment and then may enter the human body through the food chain. The investigation showed that some precious metals play an important role in the treatment of diseases and auto-immunity (1). For instance, the compounds containing Pt and Au have been used for the treatment of cancerous tumors and rheumatoid arthritis (2). However, it was also reported that most of the precious metal complexes are soluble and pose potential toxic effects to human beings (3-5). The haloge-nated Pt and Pd compounds ($PdCl_2$ and $PtCl_2$) are highly potent, sensitizing substances causing asthma, nausea, hair loss, spontaneous abortion, dermatitis, and other serious health problems (6, 7). Conse-quently, it is crucial to determine trace/ultra-trace precious metals in biological and environmental samples to study the problems with respect to environmental pollution and public health.

The determination of precious metals in biological and environmental samples is challenging due to their low concentrations and the complex effects of the matrix (8, 9). A number of sophisticated detection techniques have been used for the determination of precious metals in various samples, such as stripping voltammetry (SV), atomic absorption spectrometry (AAS), neutron activation analysis (NAA), inductively coupled plasma atomic emission spectrometry (ICP-AES), and inductively coupled plasma mass spectrometry (ICP-MS) (10-14). SV is one of the most sensitive techniques for measuring individual precious metals, but this technique does not allow for multi-element analysis of these metals. In some cases, AAS was applied for the determination of precious metals, but it suffers greatly from a limited matrix tolerance. NAA is a powerful method for the analysis of precious metals, but it is not suitable for routine analysis owing to the complexity and cost of the required instrumentation. Although ICP-AES provides fast and multielement analysis, it often does not entirely meet the requirements for the determination of precious metals, especially at the nanogram per gram (ng g^{-1}) or lower concentration levels. The most favorable choice for precious metals analysis is ICP-MS because of its high sensitivity, wide linear range, and rapid multielement detection capability. However, matrix interferences originating from relatively complex samples can affect the measurement

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signal to a large extent. As a consequence, an effective separation and preconcentration procedure is usually necessary prior to metal determinations by these techniques.

The most widely used techniques for the separation and preconcentration of precious metals includes fire assay, coprecipitation, solvent extraction, cloud point extraction, ion-exchange, adsorption, solid phase extraction, and liquid phase microextraction (15-22). In recent years, much research has been oriented towards the development of efficient, cost-effective, simplified, and miniaturized sample preparation methods (23-26). Among them, dispersive micro-solid phase extraction (DMSPE) has received increased attention owing to its merits of reduced solvent consumption, less adsorbent usage, short extraction time, and high extraction efficiency (27-31). For this technique, the type of sorbent used is very important because its performance is directly related to its effectiveness. Hence, current research in DMSPE mainly focuses on the preparation of novel adsorption materials and the evaluation of their characteristics (32). It is worth noting that magnetic nanomaterials have shown excellent application potential as adsorbents because of their excellent magnetic characteristic, ease of surface modification, large surface area, and high adsorption activity. Some magnetic nanomaterials have been proposed for the preconcentration and separation of inorganic and organic substances in DMSPE (33-38).

Magnetic $ZnFe_2O_4$ nanotubes (ZFONTs) have a tubular structure with many pores on their wall. This unique structure of the ZFONTs results in a large increase in the specific surface area and the adsorptive sites. Therefore, ZFONTs have the potential of becoming a great adsorbent for DMSPE. A literature survey revealed that no studies have so far been published where magnetic DMSPE with ZFONTs was used for the subsequent determination of trace/ultra-trace precious metals by ICP-MS (39, 40).

In the present work, ZFONTs were used as an adsorbent for DMSPE of Au(III), Pd(II), and Pt(IV) before ICP-MS detection. The factors affecting the preconcentration and determination of the analytes were optimized and selected. The proposed method was validated by the determination of trace/ultra-trace Au, Pd, and Pt in biological and environmental certified reference samples, including human hair, soil, and stream sediment.

EXPERIMENTAL

Instrumentation

An X-7 Series ICP-MS system (Thermo Fisher Scientific, USA) with a Babington nebulizer was used for the determination of Au, Pd, and Pt. The operating parameters for the ICP-MS are given in Table I. A pH meter with a combined electrode was used to control the pH values of the solutions (Thermo Fisher Scientific, USA). The mixture was sonicated using a model KQ-50E ultrasonic bath (Kunshan Ultrasonic Instrument Co., Ltd., Suzhou, P.R. China). Sample digestion was carried out in an Ethos T microwave digestion device (Milestone, Italy). A strong neodymium-iron-boron ($Nd_2Fe_{12}B$) magnet was used for the magnetic separation.

Reagents and Standard Solutions

The standard stock solutions of Au, Pd, and Pt (1.0 mg mL^{-1}) were obtained from the National Analysis Center of Iron & Steel (Beijing, P.R. China). The working solutions were prepared by stepwise dilution of the above stock solutions just before use. Unless otherwise stated, all chemicals used in this work were of analytical grade and purchased from Shanghai Reagent Factory (Shanghai, P.R. China). High purity deionized water was obtained from the Milli-Q® A10 system (Millipore Corporation, USA) and used throughout this work. The ZFONTs were prepared and characterized in our laboratory (39). When not in use, all glassware was kept permanently full of 2.0 mol L^{-1} HNO_3 solution.

Dispersive Micro-solid Phase Extraction Procedure

A 40-mg amount of ZFONTs was added to 50 mL of sample solution at pH 3.0. Then, the mixture of sorbent and sample was sonicated with a vortex for 1.5 minutes to

TABLE I
Operating Conditions for ICP-MS

Plasma Power	1.3 kW
Plasma Argon Flow Rate	14 L min^{-1}
Auxiliary Argon Flow Rate	0.86 L min^{-1}
Nebulizer Argon Flow Rate	0.95 L min^{-1}
Sampler Orifice (nickel)	1.1 mm
Skimmer Orifice (nickel)	0.7 mm
Acquisition Mode	Peak jumping
Number of Sweep	100
Dwell Time	10 ms
Acquisition Time	40 s
Number of Measurements per Peak	3
Isotope	^{197}Au, ^{105}Pd, ^{195}Pt

facilitate the dispersion of the ZFONTs in the sample solution. After extraction, the ZFONTs were separated from the aqueous solution with the help of a permanent magnet. Subsequently, the aqueous phase was discarded, and the ZFONTs remained in the test tube. To elute the analytes, 0.5 mL of 1.5% thiourea in 0.5 moL L^{-1} HNO_3 solution was added to the test tube containing the ZFONTs, and the test tube was ultra-sonicated for 1.0 minutes. Then the sorbent was separated with the magnet again. Finally, the eluent was taken out with a pipette for ICP-MS analysis.

Sample Preparation

A 0.5 g of sample (stream sediment, soil, and human hair) was accurately weighed into PTFE digestion vessels. Then, 1.5 mL of HNO_3 (65~68%, w/w), 4.5 mL of HCl (36%~38%, w/w), and 2.0 mL of HF (40%, w/w) were immediately added to the vessels. After the first vigorous reaction had taken place, the vessels were tightly capped and placed in the digestion device. The samples were digested at 200 °C (ramp, 10 min; hold, 30 min) at a power of 1.0 kW. After cooling, the solution was transferred into a Teflon® beaker, then evaporated to low volume. The residues were dissolved with 0.1 mol L^{-1} HNO_3 solution and diluted to the desired volume. The blanks were prepared using the same procedure, but without addition of sample.

RESULTS AND DISCUSSION

Effect of Solution pH

The acidity of a sample solution plays an important role in the adsorption of the analytes. First, the adsorption depends on the metal species present in the solution. Secondly, it is related to the protonation and deprotonation of active sites on the surface of the adsorbent. Thus, the effect of pH on the adsorption of the analytes was studied in the pH range from 1.0 to 9.0. The results in Figure 1 indicate that the analytes were quantitatively recovered in the pH range of 1.0–5.0, and the recovery of the analytes decreased at pH > 5.0. Therefore, pH 3.0 was selected as the optimum condition for further experiments.

The adsorption behaviors of the analytes may be explained by the surface charge of the ZFONTs at different pH values. The pH at which the zeta potential equals zero is called the isoelectric point (IEP), and it is used to assess the adsorbent surface charge quantitatively. In the sample solution, Au(III), Pd(II), and Pt(IV) mainly exist as $AuCl_4^-$, $PdCl_4^{2-}$, and $PtCl_6^{2-}$, respectively. At pH < IEP (6.0), the surface charge of the ZFONTs is positive due to the protonation reaction. The surface of the ZFONTs with the positive charges attracts $AuCl_4^-$, $PdCl_4^{2-}$, and $PtCl_6^{2-}$, thus resulting in high recoveries. At pH > IEP (6.0), however, the surface of the ZFONTs with the negative charges repels the analytes and results in low recoveries.

Choice of Eluent

In order to desorb the target ions, the different eluents, including NaOH, HCl, HNO_3, and thiourea, were examined by this method. The results in Table II show that the recoveries of the analytes were not quantitative when NaOH, HCl, HNO_3, and thiourea were used as the sole eluent, respectively. Therefore, a mixed

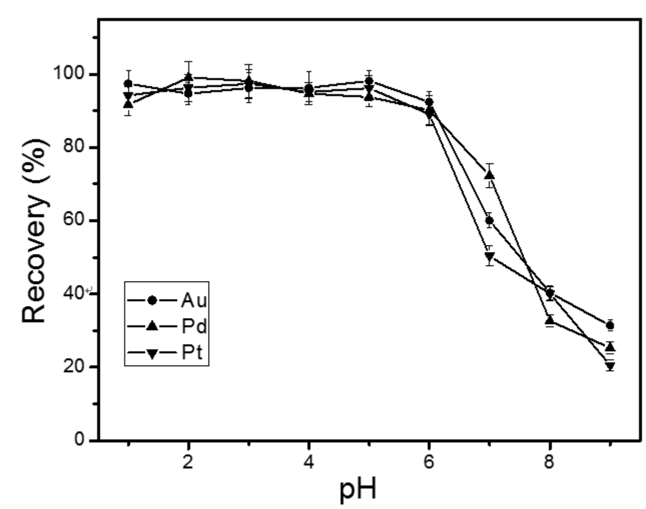

Fig. 1. Effect of pH on adsorption of analytes on ZFONTs.

TABLE II
Effect of Eluent on Recovery of Analytes

Eluent	Recovery (%) Au(III)	Pd(II)	Pt(IV)
0.5 moL L^{-1} NaOH	32.7	40.2	28.4
0.5 moL L^{-1} HCl	45.8	48.9	36.9
0.5 moL L^{-1} HNO_3	51.2	54.7	48.3
1.5% thiourea	64.7	70.2	61.7
0.5 moL L^{-1} HNO_3 + 1.5% thiourea	97.6	95.8	94.9

solution of HNO_3 and thiourea was chosen to elute the analytes since thiourea is a good sulfur donor for Au(III), Pd(II), and Pt(IV). The composition of the mixture of HNO_3 and thiourea solution was optimized for the elution of the analytes. It was found that quantitative recoveries were obtained when 1.5% thiourea in 0.5 moL L^{-1} HNO_3 solution was used as the eluent. It is pointed out here that HNO_3 is the most recommended solvent for ICP-MS determination. Hence, 1.5% thiourea in 0.5 moL L^{-1} HNO_3 solution was chosen for subsequent experiments.

Effect of Eluent Volume

In order to investigate the effect of eluent volume on the recovery of the analytes, 0.1, 0.3, 0.5, 0.8, and 1.0 mL of 1.5% thiourea in 0.5 moL L^{-1} HNO_3 solution were used to elute the target ions adsorbed on the ZFONTs, respectively. The experimental results showed that 0.5 mL eluent was enough for the complete elution of the analytes. Thus, 0.5 mL of 1.5% thiourea in 0.5 moL L^{-1} HNO_3 solution was employed in this work.

Optimization of Extraction/ Desorption Time

Extraction time is an important factor affecting the speed of extraction and is defined as the time interval between the injection of the sorbent and the application of the external magnetic field for phase separation. Thus, the effect of the extraction time under the optimized conditions was investigated in the range of 20 seconds to 3.0 minutes. The results showed that the target ions can be quantitatively recovered within 1.5 minutes. In addition, the effect of desorption time in the range of 20 seconds to 2.0 minutes was also evaluated. The results showed that 1.0 minute was sufficient for the quantitative desorption of the analytes from the sorbent.

The above facts can be attributed to the extremely large surface area between the dispersed sorbent and the aqueous phase. Thus, the adsorption/desorption process of the analytes was completed within a short time.

Influence of Adsorbent Dose

Another important parameter affecting the extraction efficiency is the amount of sorbent. For this reason, the dose of the sorbent was optimized by varying the amounts of adsorbent (10–50 mg). The results in Figure 2 show that 30 mg of adsorbent was sufficient for the quantitative recovery of the target ions (>90%). Therefore, a 40 mg amount of ZFONTs was selected for this work.

Effect of Sample Volume

The capability of extracting trace amounts of analyte from a large sample volume is another important aspect of method development. For this reason, different volumes of sample solution (10–150 mL) containing 10 ng of the target ions were extracted under the optimized conditions (Figure 3). The results of this study show that the extraction efficiency remained constant at the maximum sample volume of 100 mL. Thus, a preconcentration factor of 200 was achieved in this work based on the final volume of the desorbing solution (0.5 mL).

Effect of Potentially Interfering Ions

To evaluate the anti-interference capability of this method, the effect of common coexisting ions on the recovery of the analytes was investi-

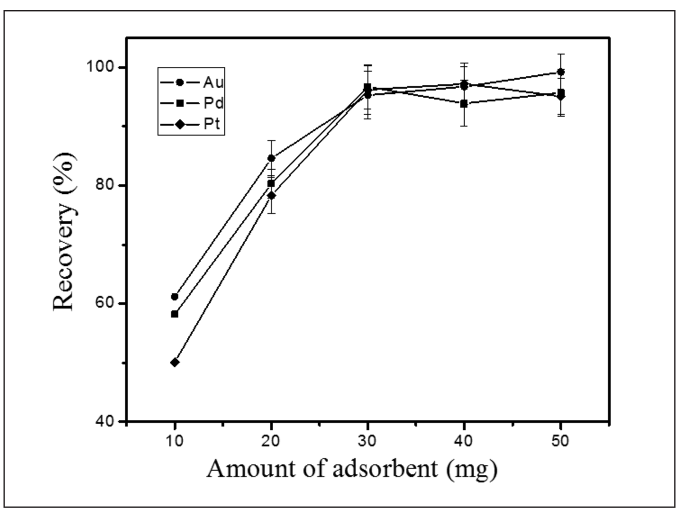

Fig. 2. Effect of adsorbent dose on recoveries of analytes.

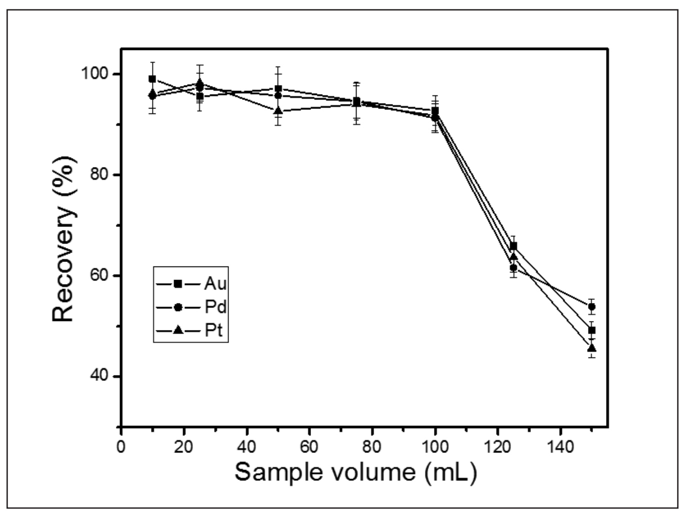

Fig. 3. Effect of sample volume on adsorption of analytes on ZFONTs.

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gated. In the experiments, solutions of 2.0 ng mL^{-1} analytes containing the added interfering ions were treated according to this procedure. When the recoveries of the target ions were kept in the range of 90%–110%, the interference caused by the co-existing ions was considered to be negligible. It can be seen from Table III that the major cations and anions do not interfere with the extraction and determination of the analytes under the selected conditions. It can be concluded that this method has a good application potential in the determination of trace/ultratrace levels of Au, Pd, and Pt in real samples.

Adsorption Capacity

The adsorption capacity, considered as one of the key factors in evaluating the performance of adsorption material, was investigated based on the method reported in the literature (41). 50 mL aliquots of sample solutions containing the analytes in the concentration range of 3.0–21 μg mL^{-1} were preconcentrated and eluted under the selected conditions. The amount of the analytes adsorbed on the ZFONTs (mg g^{-1}) at each concentration level was determined by this method. Breakthrough curves were plotted by the concentration of the analytes versus their amount adsorbed on per gram of adsorbent (Figure 4). The adsorption capacities for Au, Pd, and Pt calculated from the breakthrough curves were 12.9, 14.1, and 11.8 mg g^{-1}, respectively.

TABLE III
Effect of Coexisting Ions on Recovery of the Analytes

Coexisting Ion	Concentration[a] (mg L^{-1})
Na^+, K^+	10,000
Ca^{2+}, Mg^{2+}	5,000
Fe^{3+}, Al^{3+}	300
SO_4^{2-}, CO_3^{2-}, PO_4^{3-}	3,000
Cl^-, NO_3^-	10,000

[a] Recovery: > 90%

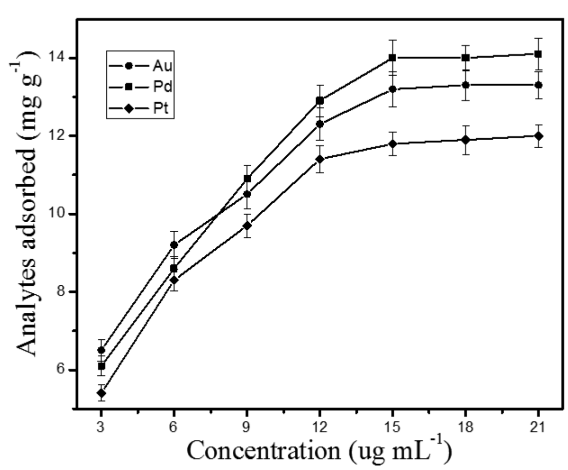

Fig. 4. Breakthrough curves of analytes on ZFONTs.

Analytical Performance

The analytical parameters of this method, including detection limits, precision, correlation coefficient, and linear range, were evaluated under the selected conditions. According to the IUPAC definition, the detection limits (DLs) of the method, defined as three times the standard deviation of the blank signal intensity, were calculated to be 0.35, 0.17, and 0.64 pg mL^{-1} for Au, Pd, and Pt, respectively. The relative standard deviations (RSDs) for nine replicate determinations of 1.0 ng L^{-1} of the analytes were 4.1% (Au), 4.9% (Pd), and 5.6% (Pt). The linear range of the calibration covered over four orders of magnitude with a correlation coefficient higher than 0.9961.

For comparison, the analytical performance of this method and similar methods found in the literature for the determination of the target ions is listed in Table IV. As can be seen, the DLs of the proposed method are lower than those reported in the literature (42-46). The RSDs are similar to other methods.

Sample Analysis

This method has been applied to the determination of Au, Pd, and Pt in biological and environmental certified reference materials, including human hair, soil and stream sediment (Institute of Geophysical and Geochemical Prospecting, Langfang, P.R. China). The analytical results are presented in Table V. As can be seen, the determined values are in good agreement with the certified values, indicating that this method is capable of analyzing trace/ultratrace levels of Au, Pd, and Pt in real samples.

CONCLUSION

In summary, magnetic ZFONTs as an adsorbent were used in DMSPE for the preconcentration and determination of trace/ultratrace levels of Au, Pd, and Pt by ICP-MS. The adsorption behavior of the analytes on the ZFONTs was investigated systematically. The analytes were quantitatively adsorbed in the pH range of 1.0–5.0, and diluted with 0.5 mL of 1.5% thiourea in 0.5 mol L^{-1} HNO_3 solution. Compared with traditional SPE, DMSPE allows close contact between the sorbent and the ana-

TABLE IV
Comparison of Analytical Performance for This Method with Others

Method	DL (pg mL^{-1}) Au	Pd	Pt	RSD (%) Au	Pd	Pt	Reference
CPE-ICP-MS	0.8	6.0	0.6	5.4	4.1	2.9	(42)
CME-ICP-MS	5.9	8.3	-	6.5	1.1	-	(43)
SPE-ICP-MS	0.46	0.26	-	5.2	2.4	-	(44)
DSPE-ICP-MS	1.7	2.5	1.9	1.2%–2.1%	1.2%–2.1%	1.2%–2.1%	(45)
MSPME-ICP-MS	4.4	-	8.6	5.9	-	7.2	(46)
DMSPE-ICP-MS	0.35	0.17	0.64	4.1	4.9	5.6	This work

CPE, cloud point extraction; CME, capillary microextraction; SPE, solid phase extraction; DSPE, Displacement solid-phase extraction; MSPME, magnetic solid-phase microextraction; DL, detection limit; RSD, relative standard deviation.

TABLE V
Analytical Results of Analytes in Certified Reference Materials

Sample	Element	Found[a] (ng g^{-1})	Certified (ng g^{-1})
Soil (GBW 07288)	Au	0.83 ± 0.07	0.9 ± 0.2
	Pd	0.28 ± 0.02	0.26 ± 0.05
	Pt	0.24 ± 0.03	0.26 ± 0.05
Human Hair (GBW 07601)	Au	2.6 ± 0.2	2.5[b]
	Pd	0.95 ± 0.08	-
	Pt[c]	Nd	-
Stream Sediment (GBW 07289)	Au	10.9 ± 0.75	10 ± 2
	Pd	2.5 ± 0.19	2.3 ± 0.2
	Pt	1.7 ± 0.2	1.6 ± 0.3

[a] Mean value ± standard deviation, n=3.
[b] Reference values.
[c] No detection.

lytes in the preconcentration step, which greatly improves the extraction efficiency. Another advantage of the magnetic ZFONTs used as the adsorbent is that the separation of the sorbent from the aqueous phase was achieved by an external magnetic field. This greatly improves the sample treatment efficiency and avoids the time-consuming column filtration/centrifugation process. Thus, the proposed method has great potential in its application for the preconcentration and determination of ultra-trace/trace levels of elements in real samples with a complex matrix.

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