



Simultaneous separation of noble metals osmium and iridium in simulated leaching of spent catalysts using nano-alumina microcolumn



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ABSTRACT

Nano- Al_2O_3 was employed as the micro-column packing material for simultaneous separation/preconcentration of osmium(Os) and iridium(Ir) species. It was found that both of Os and Ir species could be adsorbed quantitatively on nano- Al_2O_3 within a pH range of 4.0–8.0, however, only Ir(IV) could be quantitatively retained on the micro-column within a pH range of 8.0–10.0, Os(IV) were passed through the micro-column without the retention. Based on this fact, a new method of micro-column separation/preconcentration was developed for simultaneous separation of Os and Ir species. The extraction recoveries for the mentioned noble metals were more than 95%. Nano- Al_2O_3 micro-column was reused for four times without obvious significant loss of its adsorption activity for Os and Ir species. In order to validate the developed method, recovery of Os(IV) and Ir(IV) from simulated leaching solution of spent catalysts (SLSSC) was achieved successfully. The negligible uptake of Cu(II), Zn(II), Co(II), Ni(II), Cd(II), Pb(II), and Fe(III) in SLSSC was due to the high selectivity and extractability of nano- Al_2O_3 for osmium and iridium.

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1. Introduction

As precious platinum group metals (PGMs), such as Os and Ir are extensively used in automobile, catalysis, petroleum and electronics industry owing to their specific physical and chemical properties [1–4]. The natural abundances of Os and Ir in the earth crust are very low, and the natural resources for these metals are limited. The present deposits will not be enough to meet their growing demand in near future. However, millions of tons of spent electronic devices and some catalyzer waste are discarded every year [3]. These wastes contain high value Os/Ir precious metals, which indicate not only the loss of huge amounts of resources but also the threat of environmental pollution. It is important to find an effective separation process to recover these metals from high level waste solutions generated from PGM based spent catalysts and spent electronic devices [5,6].

Os and Ir elements in the same period tend to have similar chemical properties [7,8]. This makes the recovery and separation of both metals in the same solutions difficult process in hydrometallurgy. Many studies have been reported on the separation of Pt(IV), Pd(II), Au(III), Os(IV) and Rh(III) along with other platinum

metals [9–12], whereas osmium and iridium have rarely been investigated.

Several separation methods such as solvent extraction [13–18], ion exchange [19–22], sorption [23,24], precipitation [25–27], extraction chromatography [6,28–30] have been reported for the separation of PGMs. Liquid–liquid extraction is generally used for the separation and recovery of Os/Ir from spent catalysts in hydrometallurgical process. The extraction separation study of iridium, osmium and ruthenium long with other platinum metals has been carried out by various workers. However, the extraction methods suffer from one or more disadvantages such as various pre-extraction along with pre-separation steps, pre-extraction steps or co-extraction of other base metals, and are costly and time-consuming. Furthermore, large quantities of secondary waste are generated from the addition of chemical agents for precipitation and reduction in the processes.

Compared to these methods, SPE has achieved widespread use because of its simple procedure, higher pre-concentration factor, rapid phase separation and combination with different detection techniques [31,32]. Nano-materials have attracted much attention due to its high adsorption capacity, simple operation, rapid adsorption process, etc. Thus, there is a growing interest in the application of separation and pre-concentration of trace metal ions using nano-sorbent [33–35]. However, literature studies on the separation efficiency of nano-particles for Os and Ir are very limited.

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The present work deals with the evaluation of nano- Al_2O_3 micro-column for the separation and recovery of Os and Ir from SLSSC. Our study focused on the effectiveness of adsorption and separation on micro-column. The optimized experimental conditions of column separation were established.

2. Experimental method

2.1. Materials and reagents

Nano- Al_2O_3 was purchased from Beijing Nachen Nano-Meter Material Co., China and its particle size was in 10–15 nm range.

$(\text{NH}_4)_2\text{OsCl}_6$ ($\geq 99.99\%$, Os 42.5% min) was purchased from Alfa Aesar (Tianjin) Chemical Co., Ltd. A $1000 \mu\text{g mL}^{-1}$ standard stock solution of Ir(IV) in 10% HCl was purchased from National Iron and Steel Research Institute, China.

A standard stock solution of Os(IV) ($1000 \mu\text{g mL}^{-1}$) was prepared by dissolving 0.2308 g $(\text{NH}_4)_2\text{OsCl}_6$ in 1.2 mol L^{-1} hydrochloric acid and then diluting to a 100 mL with 1.2 mol L^{-1} hydrochloric acid in brown glass flask volumetric. The standard stock solutions of Os(IV) and Ir(IV) were diluted successively to the required concentration in the experiment by deionized water.

All the other reagents including hydrochloric acid, nitric acid and sodium hydroxide, were of analytical reagent grade and obtained from Sinopharm Chemical Reagent Co., Ltd. without any further purified. The standard stock solutions of all of the other metal ions were obtained by dissolving their chloride or nitrate salts ($\geq 99.99\%$) and used to investigate the adsorption selectivity. Deionized water was used throughout the experiments.

2.2. Apparatus

A Cary 5000 UV–vis–near-IR spectrophotometer (Varian) was used for the determination of Os(IV) and Ir(IV) ions. A Mettler X Series II inductively coupled plasma-mass spectrometry (ICP-MS; Thermo Scientific) was used for the determination low concentrations of Os(IV), Ir(IV) and other metal ions. A pHSJ-4F pH meter Instrument (Shanghai LEICI Co. Ltd.) was used for pH measurement. HZQ-Q thermostat oscillator (HDL APPARATUS Co. Ltd.) was used in static batch experiments. A Malvern Zetasizer Nano-ZS particle analyzer (Malvern, UK) was used to determine the zeta potential of adsorbents. A constant flow peristaltic pump with computer displaying and fraction collector (Shanghai Huxi Analysis Instrument Factory CO., Ltd.) was used in separation and pre-concentration process.

2.3. Adsorption of osmium and iridium on the nano- Al_2O_3

Adsorption tests were carried out to examine the adsorption behavior of Os(IV)/Ir(IV) on the nano- Al_2O_3 . Adsorption of Os(IV)/Ir(IV) as a function of pH was first examined in a series of experiments where the initial Os(IV)/Ir(IV) concentration was maintained constant ($5 \mu\text{g mL}^{-1}$) at varying pH values, and thus the optimum pH for adsorption was determined. Here the pH was changed by adding a small amount of NaOH or HCl. The adsorption tests were carried out individually for Os(IV)/Ir(IV) in 50 mL conical flasks by taking 10 mg of nano- Al_2O_3 together with 10 mL of target analytes (Os(IV)/Ir(IV) in the concentration range 0.5–30.0 $\mu\text{g mL}^{-1}$) solution. The flasks were then shaken in a batch thermostated system at $25 \pm 0.5^\circ\text{C}$, under agitation at a constant speed of 250 rpm for about 10 min to up to equilibrium. Afterward, all samples were centrifuged for 5 min at 5000 rpm and their equilibrium concentrations were measured. All experiments were performed at least twice. Average values are reported. Solutes and

adsorbent blanks were simultaneously run for control under the same conditions.

2.4. Solid phase extraction column prepared

The column was a simple glass tube with an inner diameter of 6.0 mm and a length of 20 cm. A known quantity of the sorbent (10 mg) was mixed with an optimum quality ratio of 1:10 for nano- Al_2O_3 to glass beads, and then the mixture was dried in a muffle furnace at 473 K for 2 h. The glass beads were placed at the column base for providing a uniform inlet flow and good targets solution distribution into the column. The nano- Al_2O_3 loaded on glass beads was packed into the column, the glass wool were kept at the bottom and at the top of the column, respectively, then the column was conditioned with deionized water for 1 h, the solution was pumped in an up-flow mode through packed-bed column by a peristaltic pump and collected by an automatically collection at room temperature (Schematic Diagram of the Column Experiment see in our previous work [36]). Then, the micro-column was conditioned to the desired pH values with HCl and NaOH solutions.

2.5. Column separation

2.5.1. Separation of Os(IV) and Ir(IV) during adsorption

The pH value of the mixture solution was adjusted to 9.5, and passed through the nano- Al_2O_3 packed micro-column by using a peristaltic pump at flow rate of 0.1 mL min^{-1} . Os(IV), which could not be retained by the column, passed directly through the column, and effluent solution containing Os(IV) from column was collected; while Ir(IV) retained by the column, was eluted with 2 mol L^{-1} HNO_3 eluent solution at flow rate of 0.05 mL min^{-1} .

2.5.2. Separation of Os(IV) and Ir(IV) during desorption

The pH value of the mixture solution containing 5 $\mu\text{g mL}^{-1}$ Os(IV) and Ir(IV) was adjusted to 6.0, and passed through the nano- Al_2O_3 packed micro-column by using a peristaltic pump at a desired flow rate of 0.1 mL min^{-1} . Both of Os(IV) and Ir(IV) could be retained by the column, the first, Os(IV) was eluted with 0.1 mol L^{-1} NaOH eluent solution at flow rate of 0.05 mL min^{-1} , the column was conditioned with deionized water, and then Ir(IV) retained on column were eluted by 2 mol L^{-1} HNO_3 . Finally, the concentrations of the separated Os(IV) and Ir(IV) in the final solutions were determined.

2.6. Simulated leaching solution of spent catalysts

SLSSC solution was prepared by dissolving metal salts in 1.2 mol L^{-1} HCl and diluting the stock solution to appropriate concentration of metal ions. The concentrations of the metal ions present in SLSSC solution consisted of 2.63×10^{-5} mol L^{-1} Os(IV) and 2.60×10^{-5} mol L^{-1} Ir(IV) and 0.02 mol L^{-1} Cu(II), Zn(II), Co(II), Ni(II), Cd(II), Pb(II) and Fe(III).

3. Results and discussion

3.1. Static batch procedures

3.1.1. Effect of pH and contact time on adsorption equilibrium

Os and Ir on nano- Al_2O_3 were studied according to the general procedure. Fig. 1A is the effect of pH on the adsorption of inorganic Os and Ir species on nano- Al_2O_3 . It can be seen that both of Os and Ir species are adsorbed quantitatively on nano- Al_2O_3 within a pH range of 4.0–8.0. When $\text{pH} > 7$, the adsorption percentage of Os(IV) decreased rapidly and almost no adsorption was observed

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