



## Review

## Sorbent materials for separation and preconcentration of gold in environmental and geological samples – A review

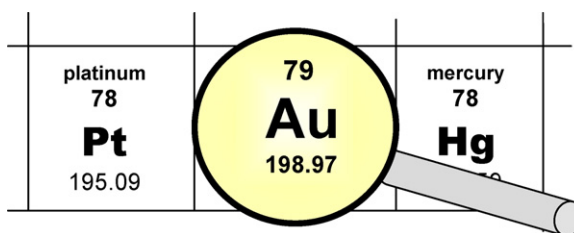
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## HIGHLIGHTS

- ▶ Gold determination requires very often preconcentration and separation.
- ▶ Solid phase extraction technique offers for this purpose high enrichment factor, rapid phase separation and the ability of combination with different detection techniques.
- ▶ The recent developments in this area were presented and discussed.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Determination of gold in environmental and geological samples requires very often preconcentration and separation due to the high concentration of interfering matrix components and the low content of this metal. Solid phase extraction technique with different kind of solid sorbents offers for this purpose high enrichment factor, rapid phase separation and the ability of combination with different detection techniques. It can be easily implemented and controlled in flow systems, The recent developments in this area are presented and discussed.

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**Krystyna Pyrzynska** received her MSc and PhD in chemistry from the University of Warsaw, Poland. Now she is working in the Laboratory of Flow Analysis and Chromatography, University of Warsaw. Her research interest is focused on the application of solid sorbents for preconcentration and separation purposes, on-line systems for determination of metal ions and speciation of metal ions in environmental samples.

## 1. Introduction

Gold is one of the most important noble metals due to its wide applications in industry as well as economic activity. Gold colloids have fascinated scientists for over a century and are now heavily utilized in chemistry, biology and medicine [1,2]. Different Au(I) and Au(III) compounds have been used as therapeutic agents to treat a wide variety of rheumatic diseases [3]. The concentration of gold in environmental samples is extremely low, as  $4 \text{ ng g}^{-1}$  and  $1 \text{ ng g}^{-1}$  were reported for basic rocks and soils [4]. Trace amount of gold can be found in some wastewaters as a result of mining, electroplating industries or electronic manufacturing. The values of  $0.05 \text{ ng mL}^{-1}$  and  $0.2 \text{ ng mL}^{-1}$  were found in seawater and river water, respectively [4].

The determination of gold in environmental and biological samples is difficult for the high concentration of interfering matrix components and the low content of this metal in the most of samples [5]. In order to solve these problems conventional separation techniques have been often exploited. Among different separation and preconcentration techniques, batch and column approaches in which gold ions are sorbed on different water-insoluble solid materials and further eluted with acids or complexing reagents have been widely used [6,7]. Solid phase extraction (SPE) offers a number of important advantages in comparison with classical liquid–liquid extraction, such as reduced organic solvents usage and exposure, high enrichment factor, rapid phase separation and the ability of combination with different detection techniques. Moreover, it can be easily implemented and controlled in flow systems. Column sorption is mainly applied to enhance the sensitivity of FAAS and ICP OES and to separate the analytes from potentially interfering matrix components, particularly for ETAAS and ICP MS detection.

The mechanism of sorption depends on the nature of a given sorbent and may include simple adsorption, chelation or ion-exchange. Adsorption occurs through van der Waals forces or hydrophobic interactions, which occurs when the solid sorbent is highly nonpolar. The most common materials of this type include octadecylsilica and styrene-divinylbenzene copolymers that provide additional  $\pi$ – $\pi$  interactions when Au(III) is complexed with ligands containing  $\pi$ -electrons. The presence of appropriate functional groups in solid matrices is needed for ion-exchange or chelation. Binding of gold traces to these groups is dependent of their nature as well as the buffering conditions. The choice of solid material for enrichment and removal of gold ions should be based on the content of the analyte, sample matrix and technique for final detection, whereas higher enrichment factors can be obtained under adequate experimental conditions (time of loading sample, sorbent mass, volume of eluent).

The purpose of this paper is to present an up-to-date overview, focused on the analytical applications of different kinds of solid materials for the separation and preconcentration of gold from environmental and geological samples. The papers published over

the last five years are discussed in more detail. Earlier developments were presented in the reviews [5,7,8].

## 2. Bonded-phase silica sorbents

Silica chemically bonded with various groups has been the most common material to SPE. These groups can be classified according to their most significant properties (non-polar, polar, or ion-exchange) Non polar sorbents contain octadecyl ( $\text{C}_{18}$ ), octyl ( $\text{C}_8$ ), ethyl ( $\text{C}_2$ ) and phenyl groups, while aminopropyl, cyanopropyl) and diol groups are present in polar sorbents. Their interaction mechanisms are mainly based on hydrophobic or polar interactions, but often more than one mechanism is responsible for isolate retention. Ion exchange interactions are utilised with sorbents containing ionic groups, mainly quaternary amines (SAX) or sulfonic groups (SCX). However, silica-based sorbents are unstable at pH extremes ( $2 > \text{pH} > 10$ ) and have relatively low capacity.

The bonded-silica sorbent may be packed in different formats: filled microcolumns, cartridges or discs [6,7,9]. A variety of these phases are commercially available in the format of cartridges. Extraction can be also performed with membrane disks containing  $\text{C}_{18}$ -bonded silica ( $8 \mu\text{m}$  particles) on polytetrafluoroethylene or glass fiber supports. The typical composition of the disc is 80–90% (w/w)  $\text{C}_{18}$  or  $\text{C}_8$  and 10–20% support, the discs dimensions are 47 mm in diameter and 0.5 mm thick [10]. Disc provide shorter sample processing time on account of their larger cross-sectional area and decreased pressure drop, allowing higher sample flow rates. This is important for environmental samples, where larger sample volumes are usually employed to achieve adequate detection limits.

In the last few years, hybrid mesoporous solids based on silica have shown remarkable physical and chemical properties, which can be exploited in analytical determinations [11]. The three solids obtained from mesoporous silica and functionalized with 3-aminopropyl, *N*-[2-aminoethyl]-3-aminopropyl and 3-mercaptopropyl groups have been examined for preconcentration purposes in the determination of traces of Au(III) [12]. For the first two materials maximum sorption capacity were obtained at pH 8–9 ( $1321.7$  and  $843.0 \text{ mg g}^{-1}$ , respectively). The highest sorption capacity of sorbent with thiol groups was observed at pH 1–4 ( $783.9 \text{ mg g}^{-1}$ ) due to the electrostatic interactions of the negatively charged  $\text{AuCl}_4^-$  species and the protonated silanols, plus covalent interactions between the gold metallic centre and the functional groups. Sorbent obtained by grafting aminopropyl on the surface of mesoporous silica MCM-41 and MCM-48 for extraction, preconcentration and determination of trace amounts of gold to FAAS have been proposed by Ebrahimzadeh et al. [13]. For both materials quantitative recovery of Au(III) was obtained in the pH range of 2–4. Sorption of gold ions from strongly acidic solutions is an advantage because strong acids are usually used to decompose many materials including gold. However, the proposed sorbents have quite low sorption capacity of  $281.7$  and  $305.3 \text{ mg g}^{-1}$  of Au(III) [13]. The enrichment factor was 278 using the maximum sample volume of 2500 mL. When the volume of eluent ( $0.1 \text{ mol L}^{-1}$  thiourea prepared in  $3 \text{ mol L}^{-1}$  HCl) was reduced by boiling to 3 mL, higher value of 833 was obtained.

Several modifications were used to immobilize different compounds on the surface of classical silica-base sorbents to increase their selectivity [14]. For preconcentration of gold thiourea derivatives [15,16], tioethers [17], 8-hydroxyquinoline [18] and flavonoid morin [19] have been proposed. The adsorption potential of nanometer  $\text{TiO}_2$  immobilized on silica gel was evaluated by Liu and Liang [20]. Nanoparticulate metal oxides exhibit intrinsic surface reactivity and high surface areas, and can chemisorb many substances. Quantitative adsorption of Au(III) on immobilized

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