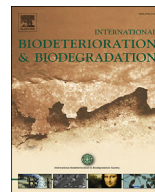




Contents lists available at ScienceDirect

International Biodeterioration & Biodegradation

journal homepage: www.elsevier.com/locate/ibiod

A review of antimony (Sb) isotopes analytical methods and application in environmental systems

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ARTICLE INFO

Article history:

Received 15 May 2016

Received in revised form

1 November 2016

Accepted 2 January 2017

Available online xxx

Keywords:

Sb isotopes

MC-ICP-MS

Pre-concentration and separation

Isotope fractionation

ABSTRACT

Antimony (Sb) is a metalloid element with potential toxicity and carcinogenicity, and is ubiquitous in our living environment. Sb pollution has become a serious global problem, which has been induced by combustion of fossil fuel and Sb-containing waste as well as mining activities. However, Sb geochemical cycle in environment systems and its inherent hazards to human and ecosystem health are poorly known. Sb isotope technology provides a novel method to counter this challenge. Sb isotopic ratio could not be measured precisely until the introduction of the multiple-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) at the beginning of the 20th century. This paper systematically reviewed the analytical methods of Sb isotopes, including pre-concentration and separation methods of thiol cotton fiber (TCF) and ion-exchange chromatography, measurement techniques of MC-ICP-MS with hydride generation (HG) and metal doping. Additionally, the Sb isotope fractionation mechanism was summarized in biological, reduction, adsorption, evaporation, precipitation and mixing processes. The Sb isotope application research status is also reviewed, such as illustrating Sb sources and its characteristics of migration and transformation in hydrologic system. Finally, future prospects for Sb isotopes in environmental systems have been proposed, which will promote the advances in the study of Sb pollution and its prevention significantly.

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1. Introduction

Antimony (Sb), situated in group 5a of the Periodic Table of the Elements with atomic number 51, was verified in 1980. Sb is a chalcophile element because of its strong affinity to sulfur and oxygen. Abundance of Sb in lithosphere is 0.2 mg/kg, mainly in the form of oxides and sulfides (Filella et al., 2002a). Antimony and its compounds are widely used in making ceramics, glass, batteries, paint, firework materials, ammunition and fire-retardant. Sb is a metalloid element with potential toxicity and carcinogenicity, and it is widespread in the environment (Sundar and Chakravarty, 2010; Winship, 1987). The chemical property of Sb is similar to arsenic (As) (Andrewes et al., 2000; Gebel, 1997), and it commonly exhibits four formal oxidation states: (V), (III), (0) and (-III) in the environment (Sigel et al., 2005). Sb and its compounds were considered as

pollutants of priority interest by the European Union (Council of the European Communities, 1976) and the Environmental Protection Agency of the United States (USEPA, 1979), and Sb is also on the list of hazardous substances under the Basel convention concerning the restriction of transfer of hazardous waste across borders because of its toxicity and long distance transportation (Filella et al., 2002a).

There are two stable isotopes of Sb, ¹²¹Sb and ¹²³Sb (Kathawa et al., 2013), with abundance of 57.213% and 42.787%, respectively (Berglund and Wieser, 2011). Before the 1990s, studies of Sb were focused on the abundance (Kusch et al., 1937; White and Cameron, 1948) because Sb isotopic ratio could not be measured precisely. To analyze Sb isotopes, the difficulties were as follows: (1) Sb concentrations in geological samples and seawater are low at less than 100 ng/g and 300 ng/g, respectively (Rouxel et al., 2003); (2) Little mass difference of only 1.6% between ¹²¹Sb and ¹²³Sb indicates that pre-concentration and separation of Sb are necessary prior to measure using MC-ICP-MS; (3) Because ¹²³Sb has isobaric ¹²³Te, it is essential to separate Sb from Te, unfortunately, they could not be

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measured by stable isotope ratio mass spectrometry (IRMS) such as MAT261; (4) Sb, especially Sb(III), is rather volatile (Klein et al., 2009), inducing the loss of total Sb in the evaporation process; (5) Sb isotope fractionation can occur in the purification procedure.

Sb isotopic ratio could not be measured precisely until the introduction of the MC-ICP-MS at the beginning of the 20th century. Rouxel et al. (2003) determined the Sb isotopes in high precision and observed the Sb isotope fractionation for the first time, and forecasted that Sb isotopes could be extremely useful tracers to reveal the natural processes and paleoredox in oceanic system (Rouxel et al., 2003). Since then, Sb isotope composition and fractionation were understood. With the development of MC-ICP-MS technique, Sb isotopes in environmental systems has been further studied.

In this paper, Sb isotope analytical methods as well as fractionation and application has been summarized, with the hope that geochemical cycle of Sb isotopes will be further studied in the future, which will promote the advances in the study of Sb pollution and its prevention significantly.

2. Sb isotope analytical methods

2.1. Pre-concentration and separation

Because most natural samples have a complex chemical composition, the matrix effects usually occur when analyzing Sb isotopes with MC-ICP-MS. The matrix elements, in particular isobaric Te, can reduce the ionization efficiency of Sb, interfere with the analytical signal, and produce unpredictable mass fractionation. Hence, separation of matrices is required for precise and accurate Sb isotope data. In addition, Sb concentrations in natural samples are low, and pre-concentration and separation procedure can cause significant Sb isotope fractionation if yield of Sb is low. Therefore, it is critical to achieve excellent pre-concentration and separation of matrix elements and high recovery of Sb.

Generally, the methods of thiol cotton fiber (TCF) and ion-exchange chromatography are used for Sb pre-concentration and separation. In 1971, Nishi and Horimoto developed the TCF that was used to pre-concentrate trace methyl mercury in water. TCF is prepared by impregnating excellent fat-free cotton in the mixing solution with mercaptoacetic acid, acetic anhydride, acetic acid and concentrated sulphuric acid after being cooled, washed and dried (Marin et al., 2001; Yu et al., 2001, 2002). Commonly used resins include cation exchange resin such as Biorad AG50-X8 (Rouxel et al., 2003) and Dowex AG50-X8 (Degryse et al., 2015; Lobo et al., 2014, 2013, 2012), and anion exchange resin Amberlite IRA 743 (Degryse et al., 2015; Lobo et al., 2014, 2013, 2012). Here, we summarize the pre-concentration and separation procedures of Sb described in recent literature.

About the method of TCF, Rouxel et al. (2003) used cation exchange resin and TCF to purify Sb in seawater, mantle-derived rocks, various environmental samples, deep-sea sediments and hydrothermal sulfides (Table 1). After acid digestion and some chemical procedure, initially, a cation exchange chromatographic column filled with 2.5 mL of Biorad AG50-X8 resin was required for the removal of transitional metals such as K, Ca, Fe, Zn, Pb, Ni, and Co in solution, whereas Sb(V) and other anions (such as sulfate, chlorides and Sn, Se, Ge, As, Te) passed through the column. Then Sb(V) was reduced to Sb(III) by adding 0.5 mL of 10% KI solution stabilized in 10% ascorbic acid because TCF only adsorbs ions in their lower oxidation states (Yu et al., 2002), and the reduction was enhanced by allowing 24-h settling time. Quantitative reduction can be achieved within several minutes by heating the solution at 90 °C in this chemical procedure. Thereafter, after the reduction step, a column filled with washed and conditioned TCF was

required for the separation of other anions. Finally, Sb was eluted with 2.5 mL of 6 M HCl and additional KI and ascorbic acid were added to prevent back oxidation of Sb(III) for hydride generation analysis and MC-ICP-MS measurement. In this chemical preparation, the hydride-forming elements, such as Se, Se, Ge, As, in particular Te are retained in the TCF, and the reducing agent of KI-ascorbic acid mixed solution improves the separation efficiency of Sb and Te. Using this method, the recovery of antimony was 82%. Almost ten years later, Asaoka et al. (2011) proposed a new antimony pre-concentration and separation method for isotope analysis of Sb using a modified TCF (Table 1). In comparison with previous method, the reducing, separation and evaporation conditions were improved in this new method (Fig. 1). The sample was reduced firstly without cation exchange chromatographic column, and 0.5 w/v% KI-ascorbic acid mixed solution was used for Sb reduction with the time of three hours. After the solution passed through the TCF column, 2.5 M HCl was used for completely separation of Sb and Sn, and Sb was eluted with 5 mL of 6 M HCl finally. More specially, after Sb collection, Sb(III) needed to be oxidized to Sb(V) before isotope analysis. 3.5 w/v% H₂O₂ and 95 °C was selected for the oxidation and evaporation procedures, respectively. The final Sb sample solution was converted into 2% HNO₃ prior to isotope analysis using another MC-ICP-MS measurement method. Comparison of the previous TCF method indicates that Sb isotope fractionation is improved in this modified TCF method with the Sb recovery of 99.5 ± 3.6% (n = 3) and blank values < 0.1 ng (Table 1), and the new method is more simple and fast. Tanimizu et al. (2011) used the modified TCF method for the pre-concentration and separation of Sb in stibnite and mine drainage water from abandoned Sb mine (Table 1). Recently, based on the modified TCF method, Resongles et al. (2015) developed a novel method using thiol-cellulose power (TCP) for pre-concentration and separation of Sb in river waters affected by mining activities (Table 1). TCP was prepared using cellulose powder instead of fat-free cotton. Specially, an adequate separation of Sb was obtained after TCP transfer into a centrifugation tube using 3 mL of 6 M HCl followed by ultrasound treatment (15 min), then centrifugation (20 min) and supernatant collection. After the extraction of Sb, the sample solution was determined without oxidation procedure using similar analytical method like Rouxel et al. (2003) for isotope analysis. Using this method, the recovery of Sb was 96 ± 2% (n = 3), which is close to modified TCF method (Asaoka et al., 2011), while is better than the primary TCF method (Rouxel et al., 2003).

As for the method of ion-exchange chromatography, Lobo et al. (2012, 2013; 2014) and Degryse et al. (2015) used cation and anion exchange resins for the pre-concentration and separation of Sb in stibnite and ancient glass successively (Table 1). In a first step, Sb sample solution was loaded onto a column packed with 1 mL of Dowex AG50-X8 resin for the removal of matrix interfering elements such as K, Ca, Cu, Pb, and Fe, and Sb was retrieved with ultrapure water. Thereafter, the solution was loaded onto a column packed with 0.7 mL of 16–50 mesh Amberlite IRA 743 resin for the removal of other interfering elements such as As, Ge, Al etc., and Sb was eluted with 14.5 mL of mixed solution consisting of 0.5 M NH₄OH, 1M NH₄OH and ultrapure water. In this pre-concentration and separation procedure, the recoveries of Sb were more than 94% after cation exchange chromatography, whereas the recoveries of Sb were only more than 77% after anion exchange chromatography. Using this method, the sample solution neither needs to be reduced from Sb(V) to Sb(III) nor needs to be oxidized from Sb(III) to Sb(V), thus limiting the risk of artificial isotope because the reduction step can induce an instant isotope fractionation of 0.9‰ (Rouxel et al., 2003). Compared with the method of TCF, the method of ion-exchange chromatography seems to be more simple and fast for pre-concentration and separation of antimony. However, it can be

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