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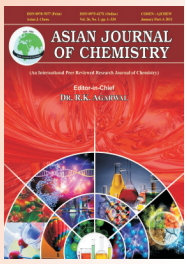

Study on Sample Pretreatment of Sediments by High Temperature and High Pressure Digestion with Bomb

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A new fast pretreatment method for sediments which digested on high temperature and high pressure ambient in bomb was proposed. The influence of the weight of sample, the species and dosage of solvents, digested time and temperature on digestion ability of sediments were studied. The results showed that the digestion effect was best when the weight of sample was nearly 0.010 g, the solvent was 0.5 mL HCl + 1.5 mL HNO_3, digested temperature and time were 160 °C and 6 h, respectively.

Keywords: Sediment, High temperature and high pressure, Bomb, Sample pretreatment.

INTRODUCTION

In recent years, with the rapid development of industry and economy, a large number of domestic wastewater, industrial sewage and heavy precipitation which are produced by pollutants into the atmosphere through dry and wet deposition, enter into the Lake water systems directly, which lead to an increased pollution of soil and water body and a growing risk for heavy metal uptake by human. Many studies[1-3] indicated that industrialization and urbanization had caused the elevated contamination of heavy metals in soils and waters. Then, a large number of heavy metals will be concentrated in Lake water systems through cycle and reach to a balance among the bottom sediments, surface water, pore water and the overlying water, which will cause influence on the Lake water quality. Finally, the elevated heavy metals in the soils and waters can pose a risk to human health as a result of entering the food chain through direct ingestion of dust or the ingestion of plants and water[4]. Therefore, research into the content of heavy metals in the water ecological system and their likely effects on the human health have important significance for water quality safety surveillance control and evaluation of heavy metal elements on the influence of the water[5-7].

Usually, the pollution degree of water quality detection is based on the contents of harmful elements, especially heavy metals elements, in Lake water systems. The HCl-HNO_3-$HClO_4$ digestion method at ambient pressure for extraction of heavy metals in sediments is often used[8], but mercury and arsenic will be loss if traditional wet digestion method is used to dispose samples due to these both elements are volatile, which will lead to a certain error for determination results. And traditional wet digestion method has many disadvantages, such as longer operating time, more labor consumption, complicated operation and new potential contamination in the transformation process[9]. The hydrofluoric acid will be added into the process, which can not use glass apparatus, consequently, experimental difficulty and risk will be increased. Therefore, sealed digestion method becomes the preferred treatment method for determination of some volatile elements and the high temperature and high pressure digestion with bomb has been widely used in the environment and the geological mineral measurement of many elements because of its many advantages, such as, needing a small amount of acid (the capacity is just 10 mL), strong compression performance (the internal pressure can reach to 12 atm.), digestion of high efficiency, environmental protection, avoiding the loss of volatile elements, don't need to hard equipment and specialized technical personnel *etc*.[10]. For example, Jin *et al*.[11] used high pressure sealed Teflon bomb to decompose sock samples and then determined 43 trace elements in samples. Hu *et al*.[12] reported the determination of Sb and Bi in 24 international geological reference materials by using pressurized acid digestion-ICP-MS.

In this paper, certified reference sediment materials (GBW07309) was selected as real sample to carry out the sample digestion condition experiment. And the weight of

sample, the species and dosage of solvents, digested time, digested temperature were identified in present study. One condition on the influence of digestion ability of sediments was studied through fixing other conditions and changing this one particular condition. 3, 6, 8, 10, 12, 16, 20 and 24 h were selected as digested time; 100, 120, 140, 160 and 180 °C were selected as digested temperature; 1, 2 and 3 mL were selected as solvent amount. Hydrochloric acid, nitric acid, hydrochloric acid + hydrogen peroxide, aqua regia and inverse aqua regia were selected as solvents.

EXPERIMENTAL

The reagents used in this study were better of analytical reagent grade. Concentrated HCl, HNO_3 and HF were all BV-III reagent grade and purchased from Beijing Institute of Chemical Reagent (Beijing, China), H_2O_2 was purchased from Beijing Chemical Factory (Beijing, China). Cu, Cr, As, Cd and Hg stock solution of 1 mg mL^{-1} were supplied by National Analytical Test Center of Nonferrous Metal and Electronic Material (Beijing, China). All analytical standard solutions were prepared by diluting the stock solution to the required concentration just before use. Ultrapure water of 18.2 MΩ cm produced from the Milli-Q Plus system (Millipore, Bedford, MA, USA) was used for preparing all solutions. Certified reference materials of sediment (GBW07309) was obtained from the Institute of Geophysical and Geochemical Exploration, Ministry of Geology and Mineral Resources (Langfang, China).

An inductively coupled plasma mass spectrometer (ICP-MS, X series II, Thermo Fisher Corp., USA) was used to detect the heavy metals, the optimal instrumental operating parameters were described in Table-1. And the standard working curves were established by external standard method with indium as internal standard and $r^2 > 0.99$ for all elements.

TABLE-1
INSTRUMENTAL OPERATING PARAMETERS

Parameters	Settings
RF power	1350 W
Nebulizer gas flow rate	0.9 L min^{-1}
Cooling gas flow rate	13 L min^{-1}
Auxiliary gas flow rate	0.8 L min^{-1}
Sampling cone	1 mm (Platinum)
Skimmer cone	0.7 mm (Platinum)
Nebulizer system	concentric glass nebulizer
Sampling depth/step	160
Resolution	125 amu
Isotopes monitored	^{63}Cu, ^{52}Cr, ^{75}As, ^{111}Cd, ^{202}Hg, ^{208}Pb

General procedure of sample treatment: Certified reference materials of sediment samples nearly 0.010 g were quantified by electro-balance which has precision of 0.0001 g, then they were added into the internal pot of Bomb (Tianjin, China) with volume of 10 mL and the internal pots were made by polytetrafluoroethene and different solvent or same solvent with different volume were added into the internal pots of bomb, then the internal cover were closed and putted the internal pots into the external pots, which were made by stainless steel, then screw thread springs were putted on the internal pots, subsequently, the external cover were closed and screwed down with spanner, finally, the total Bombs with samples were putted into Drying Box (DHG-9023A, Shanghai, China) to heat. After a period of time, the samples were poured into 10 mL clean flasks from the bomb, after washing the bomb, the solution were made up to 10 mL with pure water, this solution was then ready for the detection by ICP-MS. The blank solution was made at the same time.

RESULTS AND DISCUSSION

The experiments showed that digestion effect was not good when the sample quantity was more. The main reason was the smaller volume of Bomb's internal pot with only 10 mL, which could not contain more solvent. If the dosage of solvent was more than one quarter of internal pot volume, the danger of leak or blast would be produced. Finally, about 0.010 g was selected as the better weight of the sample and better deliquescent effect of sediment samples would be obtained.

Selection of solvent dosage: Solvent dosage is a key of sample digestion, 1, 2 and 3 mL were studied when the solvent aqua regia was constant and the temperature was constant on 170 °C. The results showed that residual mass of the standard sediment powders was most in the pot with 1 mL aqua regia, however, the pot with 3 mL aqua regia had leak, the possible reason is the more of solvent dosage, the pressure in pot at high temperature is higher, if the pressure in pot exceeds the pressure limit of internal pot, the liquid will start leakage even make the pot distortion or explosion. So, to ensure the sediment samples digest completely and as well as avoid leakage or explosion at the condition of high temperature and high pressure, 2 mL was selected as the main solvent dosage.

Selection of solvent species: Hydrochloric acid, nitric acid, hydrochloric acid + 0.5 mL hydrogen peroxide, aqua regia and inverse aqua regia as solvents for digesting the sediments were studied at constant condition of 0.010 g for weight of sample, 2 mL for the main solvents dosage, 6 h for digested time, 170 °C for digested temperature, respectively. The results showed that the digestion ability of mix acids to sediments was better than that of one single acid such as only hydrochloric acid or nitric acid. However, the internal pot with little hydrogen peroxide had a little leakage, the reasons were that hydrogen peroxide could decompose and produce activation oxygen with high energy and a large amount of air bubbles even at lower temperature, but the volume of internal pot was only 10 mL, so, the leakage would easily cause. And the effect of digestion to sediments was better when the solvent was inverse aqua regia than that of traditional aqua regia. Therefore, inverse aqua regia, that is to say 0.5 mL HCl + 1.5 mL HNO_3, was select as the optimal solvent for digestion of sediments.

Selection of digested temperature: The temperature for the influence on digestion ability were studied with constant condition of 0.010 g for weight of sample, 0.5 mL HCl + 1.5 mL HNO_3 for solvents, 6 h for digested time, respectively. The results indicated the samples would dissolve completely with not leak when the temperature was higher than 160 °C. For saving energy, 160 °C was selected as the optimal digested temperature finally.

Selection of digested time: Different digested times were investigated through fixing other conditions. The results indicated the samples would dissolve completely with weak kelly colour when the digested times was longer than 6 h, then the heavy metals in these digested solution were detected by ICP-MS and the results were in good agreement with the certified values. For digesting sample completely and saving time to batch dispose, 6 h was selected as the optimal digested time finally.

Conclusion

In this paper, we mainly studied a pretreatment method of sediment samples and used Lake water systems certified reference materials GBW07309 as sediment sample, high temperature and high pressure ambient as digested condition, airtight Bomb as digested container. And the contents of heavy metals in digested solution were detected by ICP-MS. The weight of sample, the species and dosage of solvents, digested time and digested temperature these five conditions were selected to study the influence of these pretreatment conditions on digested effect of sediment samples. After a series of experiments, the optimal digestion conditions were obtained. when the weight of certified reference lake sediment (GBW07309) was about 0.010 g, the solvent was 0.5 mL HCl + 1.5 mL HNO_3, digested temperature was 160 °C and digested time was 6 h, the digestion effect of sediment sample was best, after detection *via* ICP-MS, the values of heavy metal elements in sample were in good agreement with the certified values. The developed pretreatment method in this study can also be used for other samples with various matrix compositions, such as soil, or suitable for batch analysis due to its high sample throughput.

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