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Microwave assisted digestion followed by ICP-MS for determination of trace metals in atmospheric and lake ecosystem

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ABSTRACT

The study of trace metals in the atmosphere and lake water is important due to their critical effects on humans, aquatic animals and the geochemical balance of ecosystems. The objective of this study was to investigate the concentration of trace metals in atmospheric and lake water samples during the rainy season (before and after precipitation) between November and December 2015. Typical methods of sample preparation for trace metal determination such as cloud point extraction, solid phase extraction and dispersive liquid-liquid micro-extraction are time-consuming and difficult to perform; therefore, there is a crucial need for development of more effective sample preparation procedure. A convection microwave assisted digestion procedure for extraction of trace metals was developed for use prior to inductively couple plasma-mass spectrometric determination. The result showed that metals like zinc (133.50–419.30 $\mu\text{g}/\text{m}^3$) and aluminum (53.58–378.93 $\mu\text{g}/\text{m}^3$) had higher concentrations in atmospheric samples as compared to lake samples before precipitation. On the other hand, the concentrations of zinc, aluminum, chromium and arsenic were significantly higher in lake samples after precipitation and lower in atmospheric samples. The relationship between physicochemical parameters (pH and turbidity) and heavy metal concentrations was investigated as well. Furthermore, enrichment factor analysis indicated that anthropogenic sources such as soil dust, biomass burning and fuel combustion influenced the metal concentrations in the atmosphere.

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Introduction

The atmospheric environment contains numerous types of air pollutants from natural and/or anthropogenic sources. Trace metals, particularly heavy metals, are gradually being introduced into the environment as contaminants and pollutants, byproducts of industry and human civilization. A significant environmental compartment in the biochemical cycles of

trace metals is the atmosphere (Hu and Balasubramanian, 2003). During the last decades, environmental pollution by toxic heavy metals at trace levels has dramatically increased. Atmospheric deposition is considered to be the major source of toxic metals such as Hg, Cd, Pb and others to ecosystems. These trace metals are present in various forms in the environment (water, soil and air). There is an increasing need to determine the natural background of trace metals

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(such as toxic heavy metals) to assess global environmental pollution. These trace metals are considered some of the most serious pollutants in the natural environment due to their toxicity, persistence and bioaccumulation, because they are being retained in plants, animals and nature in general (Gleyzes et al., 2002). The monitoring of trace metals in environmental samples is a key part of analytical chemistry due to their negative influence on human and environmental health (Najafi et al., 2010). Trace metal pollution not only affects the production of crops but also influences the quality of the atmosphere as well as water bodies, and threatens the health and life of animals, humans and microorganisms (Li et al., 2013). The environmental pollution caused by trace metals is a long-term, irreversible process and can be toxic even at low concentrations.

Trace metal pollution is a growing universal problem in the aquatic environment and currently it has reached an alarming level (Ogoyi et al., 2011). Many trace metals are regarded as serious pollutants of aquatic ecosystems because of their long environmental persistence, toxicity and ability to be incorporated into food chains. There are many sources of trace metals, however the most important sources of trace metals in aquatic ecosystems are anthropogenic activities. Conversely, trace metals also arise in minor amounts naturally and may enter into an aquatic system through leaching of rocks, airborne dust, vegetation and biomass burning (Fernandez and Olalla, 2000). Their concentration usually depends on both natural and anthropogenic sources, amount of suspended particulate matter and composition, salinity, pH and redox potential (Adiana et al., 2011). In aquatic environments, trace metal concentrations are usually monitored by measuring their concentrations in water and sediments such as those in lake ecosystems, and generally are present at low levels in water and contribute to contamination and pollution (Storelli et al., 2005).

Lakes are sensitive areas due to their potential exposure to pollutants from several sources. Lake ecosystems also provide an indication of climate variation, either directly or indirectly through their catchment. Pollutants enter the water body of a lake through the connecting rivers, runoff water and from atmospheric deposition. The limited water movement within lakes influences the degree of pollution, and high concentrations of pollutants can decrease the biodiversity, as well as the physical environment surrounding the lake ecosystems (Dudgeon et al., 2006). Atmospheric deposition is one of the major sources of pollutants in a lake ecosystem (Vuorenmaa et al., 2014). The emissions from industry, motor vehicles and agricultural activities (biomass burning) are released to the atmosphere, blown by the wind; they then interact with water and fall down to the ground through wet deposition. These pollutant particulates in air fall during rain as acid rain and as fine particles (particle size $\leq 2.5 \mu\text{m}$) (Driscoll et al., 1995). These particles can bring nutrients and trace metals into aquatic ecosystems, such as lakes, during their settling down to the surface of earth. It has been noted that the deposition of sulfates and nitrates increases the acidity of a water body (Burton et al., 2013; Ding et al., 2014). Excessive amounts of trace metals from atmospheric deposition are toxic to aquatic organisms such as fish and microorganisms (Ashraf et al., 2011; Walraven et al., 2014). Trace metals deposited into lakes are usually sequestered in the layers of sediments on the bottom of the lake. Sediment quality is a good indicator of

pollution in the water column, because it tends to concentrate the heavy metals and other organic pollutants, which can be recycled into the water body by any disturbance.

Determination of trace metals in atmospheric and lake ecosystems is important for monitoring environmental pollution. In general, direct estimation of very low levels of elements in environmental samples is often impossible due to the presence of interfering compounds or the concentration of analytes being below the detection limit of instrumentation. The most commonly used analytical method for trace metal determination is the atomic absorption spectroscopy (AAS) technique, which offers multi-elemental analysis but suffers from poor sensitivity in the determination of trace metals in environmental samples like lake water and atmospheric air samples, due to their low concentrations. Different pre-concentration techniques such as cloud point extraction, solid phase extraction and the use of chelating agents before samples that are analyzed by AAS have been developed and successfully applied in the determination of trace metals (Najafi et al., 2010; Soylak and Aydin, 2011; Gentscheva et al., 2012). However, these extraction techniques and methods of analysis are restricted for determination of trace metals in samples due to the usage of toxic organic solvents for extraction, and require extensive and complicated procedures. Inductively coupled plasma-mass spectrometry (ICP-MS) is considered attractive and one of the most sensitive techniques for the multi-elemental analysis of trace metals in various environmental samples. This technique usually requires a transformation (extraction) of the samples into solution before analysis. Using ICP-MS apparatus, the detection limits for trace metals can be down to sub-ppt or picomol/L levels in simple matrix solutions. Nowadays among the various pre-concentration procedures for trace metals, digestion procedures using closed-vessel microwave sample preparation systems are used for various environmental investigations of trace metals due to their excellent capabilities for multi-element concentration with minimum selectivity and sample manipulation.

The main objective of this investigation is to establish a simple and fast method of trace metals analysis in environmental samples, including atmospheric particulates and lake water. The trace metals from atmospheric deposition and lake samples were extracted using a combination of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) with the help of 650 W microwave energy, which is faster than other reported extraction methods. This study also quantifies the effect of trace metal concentration during rain fall in lake samples around the study area.

1. Materials and methods

1.1. Reagents and reference materials

For the preparation of reagents and standards, freshly prepared ultrapure water ($18.2 \text{ M}\Omega \text{ cm}$) from a TKA smart2plus ultrapure water system (TKA, Germany) was used. Analytical grade reagent HNO_3 (Fluka, France) and H_2O_2 (Merck, Germany) were used for the digestion experiments. An ICP-MS multi-element standard was obtained from Perkin Elmer and used for calibration of the equipment. Polyethylene bottles for water

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