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Research Article

Determination of arsenic, copper and lead in the water of villages of Chalkidiki, Greece

Abstract

Arsenic, copper and lead metals are serious contaminants for human health. These metals are all toxic above a minimum concentration. In the present study twenty two water samples (eleven sites - non and acidified) from villages Nea Silata, Nea Triglia, Eleochoria, Nea Plagia, Tenedos and Sozopolis, municipality of Poligiros, prefecture of Chalkidiki, Greece were collected for quantitatively determination of arsenic, copper-65 and lead-208 by Hydride Generation - Atomic Absorption Spectroscopy (HG-AAS) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). The kinds of water were categorized as: drinking water, irrigation water and geothermal water. The results exhibited normal levels for all the three heavy metals under examination apart from geothermal water at Eleochoria village and in some cases for irrigation water at Nea Triglia and Tenedos villages for which the levels of arsenic were a little elevated. Drinking water samples from Nea Plagia and Nea Triglia villages showed lower heavy metal levels than the official Maximum Contaminant Levels (MCL) and therefore the water at these villages is considered to be potable.

Introduction

Arsenic, copper and lead are all toxic above a minimum concentration and therefore USEPA (United States Environmental Protection Agency) and European Council Directive proposed the Maximum Contaminant Level (MCL) for drinking water to be as follows: arsenic 10 ppb, copper 1,3 ppm and lead 15ppb.

Human health effects due to arsenic are characterised by skin lesions observed as melanosis and keratosis. Chronic arsenic exposure can lead to neurological, cardiovascular, respiratory effects, or cause skin, bladder and lung cancer [1-3]. The severe health implications from the high arsenic intake reported in West Bengal, Bangladesh, Taiwan and Inner Mongolia [4-6], were mainly caused by the high levels of inorganic arsenic in water.

The quantification of trace element species is a difficult task since trace elements are often present at low concentrations relative to the detection limits of analytical instrumentation. A number of methods have been employed and summarised in reviews of the scientific literature for arsenic determination, such as spectroscopy, chromatography and electrochemical methods [7-10].

Increases in copper concentration in waters and plants have resulted from industrial and domestic waste discharge, refineries, disposal of mining washing, and the use of copper as a base compound for antifouling paints [11]. In general, a daily copper intake of 1.5–2 mg is essential. But, severe oral intoxication will affect mainly the blood and kidneys. Therefore, the trace copper content in water and food must be controlled on a daily basis.

It is well known that lead (Pb), as a kind of heavy metal, is a dangerous and important environmental pollutant. Lead can cause pathophysiological changes in several organ systems including central nervous, renal, hematopoietic, and immune system [12,13]. Lead sources are mainly lead paint and dust [14,15], leaded gas [16,17] and lead in drinking water [18,19]. Among them, lead in drinking water is a very important lead source. In fact, for instance, in USA, the average national contribution of drinking water to blood lead is currently believed to be on the order of 7%–20% [20,21].

The most common methods used for trace lead determination are Flame or Graphite-Atomic Absorption Spectroscopy (F or GAAS) and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) since these methods have low detection limits [22,23].

More specifically for Greece several studies have been made for heavy metals in water of Thessaloniki [24,25], Crete and

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Cyclades [26], gulf of Thermaikos [27] and lakes of Volvi and Doirani (Grimanis 1990).

Greece has sufficient surface and underground waters but for several reasons (natural and anthropogenic) only a part of this amount is available for drinking and irrigation activities. The major natural reasons which cause difficulties in the utilisation of the greek water are: a) unevenly distribution of water in space and time, b) unevenly distribution of water demand, c) the geomorphology of Greece, d) the dependence of Northern Greece from rivers who come from neighbour countries, e) several non hydrated or with a few water resources islands of Greece. In general, Greece has good quality water but several anthropogenic activities in the last decades have started to make obvious the debasement of surface and underground waters. The greek water quality of course, does not exhibit any significant problems yet, except of a few areas.

In the present study water samples (drinking, irrigation and geothermal water) were collected from villages of Poligiros, prefecture of Chalkidiki, Greece for the first time, in order to monitor the levels of Arsenic, Copper-65 and Lead-208. The reason for choosing to determine the isotopes 65 and 208 is due to the fact that 208-Pb is the major isotope in terms of natural abundance and 65-Cu because the other copper natural isotope has a polyatomic interference.

Methodology

Sample collection

In the present study water samples from villages of municipality of Poligiros, prefecture of Chalkidiki, Greece (Figure 1) were collected for quantitatively determination of arsenic, copper and lead by Hydride Generation - Atomic Absorption Spectroscopy (HG-AAS) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). Twenty two water samples (eleven sites, non- and acidified, denoted by *) were collected from Nea Silata, Nea Triglia, Eleochoria, Nea Plagia, Tenedos and Sozopolis for the first time, on Semptember 2007. The kinds of water samples were categorised into three different types: drinking or tap water, irrigation and geothermal water.

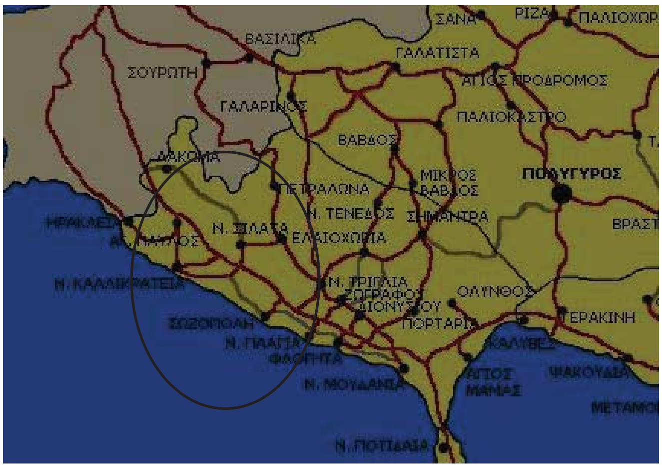

Figure 1: Map of municipality of Poligiros, prefecture of Chalkidiki showing the villages under examination into the black circle.

The flow condition of the tap and irrigation water was fast. The deapth of the drillhole from which geothermal water was pumped is 100 m and a possible source of it is leaching water from surface water. More specifically, the analytical apparatus used are (a) HG-AAS for arsenic, (b) ICP-MS for copper-65 and (c) ICP-MS for lead-208.

Sample preparation

Twenty two water samples (eleven sites-non and acidified, denoted by *) were collected in non-acidified tubes and in acidified tubes (nitric acid, BDH Aristar, UK), with a screw tap and kept to the fridge at 2 ºC until analysis. The reason for the two sets (non and acidified water samples) was to observe if acid addition stabilised the sample and see if any analyte (trace element) was lost to the container after storage. Acidification is supposed to prevent element loss.

Method of analysis for arsenic – Hydride Generation Atomic Absorption Spectroscopy

The concentration of sodium borohydride ($NaBH_4$) and hydrochloric acid (HCl) are critical to the formation of arsine (AsH_3). Therefore, these variables were tested against each other. Once the optimum conditions were found, the detection limit, precision and linearity were assessed.

The solutions were prepared with sodium borohydride from Sigma Aldrich (Reagent grade) using a Precisa 125 A calibrated balance: The solutions were prepared in 1% sodium hydroxide (NaOH) from BDH (Analar grade) in distilled water (DDW) to prevent decomposition, enabling the solutions to be kept for up to two weeks in the fridge. Acid from fisher Scientific (Primar grade) was diluted using DDW: A set of calibration standards was also prepared using a 1000 ppm standard solution. The 75 ng/ml standard was chosen to perform the optimisation study on. It decided that 1.5% $NaBH_4$ and 1M HCl were the optimum reagent concentrations. Then precision, linearity and the detection limit were assessed. A 75 ng/ml standard was run 10 times to determine the precision. The mean peak height was 172.8 ± 9.7 mm with a relative standard deviation of 5.60%. The linear range was determined by using the calibration standards. The maximum concentration used was 75 ng/ml. The correlation coefficient shows a strong linear relationship. The 10 ng/ml standard was sequentially diluted (keeping the total volume at 11 ml using DDW). At a concentration of 0.1 ng/ml, the peak was only 3 mm above the blank level, which is too close to reliably differentiate. The limit can also be determined using the following equation [28]: Limit of detection = $y_B + 3s_B$, where y_B = the analyte concentration giving a signal equal to the blank and s_B = standard deviation of the blank. The calibration curve equation ($y = 27.203x + 3.4903$) and an average blank value of 4.9 ± 0.3 mm was used to calculate the limit of detection as 0.05 + 0.4 ng/ml.

The Atomic Absorption Spectroscopy (AAS) instrument used for analysis was a PerkinElmer AAnalyst400 (PerkinElmer Instruments LLC, Shelton, CT, USA). For all elements investigated, an air/acetylene flame was used. General Atomic Absorption Spectroscopy conditions for all elements were: acetylene flow rate = 2.50 l/min; air flow rate = 10.00 l/min;

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nebulizer flow rate = 1 ml/min; burner head length = 80 mm; dwell time = 2.0 s and repeat readings = 2. Each element had a calibration run before sample analysis. The standards used for each element, were chosen based on the linear range recommended for the AAnalyst400 software (WinLab32). The calibration curves were set for linear through zero and correlations averaged at 0.99846.

Method of analysis for 208-Pb and 65-Cu - Inductively Coupled Plasma Mass Spectrometry.

The inductively coupled plasma mass spectrometry (ICP-MS) instrument used for this study was Finnigan MAT SOLA ICP-MS. The isotopes used were as follows: $^{65}Cu^{+}$, $^{57}Fe^{+}$, $^{82}Se^{+}$, $^{66}Zn^{+}$, $^{75}As^{+}$, $^{55}Mn^{+}$ and $^{98}Mo^{+}$. Instrument stability was controlled and corrected for using internal standards of 200 μg/l $^{72}Ge^{+}$, $^{115}In^{+}$ and $^{193}Ir^{+}$ were utilised for multi-element analysis. The detection limit (based on 3 sigma of the standard deviation of the blank) for most elements were below 0.7 μg/l (except Fe at 5 μg/l). The linear dynamic range of the calibration curve covered 7 orders of magnitude, so multi-element standards were run from 1 to 1000 μg/l [29-42].

Method of Validation

Quality control measurements were undertaken by replicate analysis (n=10) of two international water reference materials, namely, NIST SRM® 1643e Trace Elements in Water (National Institute of Standards and Technology, Maryland, USA) and TMDA 54.4 Trace Elements in Fortified Lake Ontario Water (National Water Research Institute, Ontario, Canada). The mean measured values and the reference range are reported in table 1. Fe, Mn, Cu, Zn, As, Se and Mo were measured as these elements are all interactive and influence the environmental fate and mobility of As. For both CRMs there is very good agreement between the mean measured levels and the certified value (Table 1).

Results and Discussion

Drinking water samples from Nea Triglia and Nea Plagia villages exhibited very low content of arsenic (below the Maximum Contaminant Level of 10 ppb for drinking water) and therefore the water at these villages is potable (range of values: 0.9 – 1.4 ppb). Irrigation water samples exhibited low levels of arsenic except of Nea Triglia and Tenedos villages where the levels of arsenic were a little elevated (range of values: 2.4 – 101.9 ppb). Also, geothermal water samples from Eleochoria village showed elevated levels of arsenic (range of values: 73.1 – 130.7 ppb) (Table 2). Geothermal water usually exhibits elevated levels of arsenic and its use as irrigation and/or drinking water has as a consequence the contamination of these kinds of water.

Drinking, irrigation and geothermal water samples from all the villages under examination exhibited low levels of copper (range: 1.0 – 110.4 ppb). Especially, drinking water from Nea Triglia and Nea Plagia villages was exhibited copper levels under the official level of 1,3 ppm for drinking water and therefore is considered to be potable (Table 2).

As it considered lead, drinking, irrigation and geothermal water samples from all the villages under examination in the present study, exhibited low levels of lead (range: 0.3 - 6.2 ppb). Drinking water from Nea Triglia and Plagia villages was exhibited lead levels under the official level of 15 ppb for drinking water and therefore is considered to be potable (Table 2).

Unfortunately, there are no previous published studies for heavy metals in water for these particular places in the municipality of Poligiros, Chalkidiki in order to compare them with the results of the present study.

Table 1: Quality Controls used for water analysis.

Element	Elemental Concentration (μg/l) (n=10)			
	CRM TMDA-54.4		CRM 1643e	
	Reference Range (95%confidence interval)	Measured Value (mean ± std dev.)	Reference Range	Measured Value (mean ± std dev.)
Iron	382 (5)	386.2 ± 59.2	98.1 ± 1.4	97.2 ± 4.5
Manganese	275.0 (2.0)	275.6 ± 33.7	39.0 ± 0.5	37.6 ± 1.3
Copper	443.0 (4.0)	446.7 ± 26.3	22.8 ± 0.3	19.5 ± 2.3
Zinc	537.0 (6.0)	534.5 ± 40.2	78.5 ± 2.2	77.9 ± 4.5
Arsenic	43.6 (0.8)	46.4 ± 1.2	60.5 ± 0.7	59.3 ± 2.3
Selenium	33.0 (0.7)	33.4 ± 2.2	12.0 ± 0.1	11.5 ± 1.3
Molybdenum	295.0 (3.0)	302.2 ± 12.2	121.4 ± 1.3	119.8 ± 12.5

Table 2: Arsenic, Copper and Lead results reported as ppb (μg/L) as determined by HG-AAS; ICP-MS-65; ICP-MS-208 respectively.

Sampling area (Kind of Water)	As (ppb)	65-Cu (ppb)	208-Pb (ppb)
Sozopolis (irrigation)	2.4	50.2	0.9
* Sozopolis (irrigation)	2.5	110.4	1.7
Nea Silata (irrigation)	6.6	6.9	0.5
* Nea Silata (irrigation)	6.4	7.2	0.5
Eleochoria (geothermal)	130.7	11.1	5.1
* Eleochoria (geothermal)	128.7	13.3	4.9
Eleochoria (geothermal)	85.4	13.3	5.8
* Eleochoria (geothermal)	73.1	15.6	4.3
Nea Triglia (irrigation)	41.0	2.2	0.5
* Nea Triglia (irrigation)	34.8	2.3	0.5
Tenedos (irrigation)	13.4	1.6	0.4
* Tenedos (irrigation)	13.4	2.2	0.4
Tenedos (irrigation)	87.5	17.7	4.9
* Tenedos (irrigation)	101.9	22.8	6.2
Nea Silata (irrigation)	6.8	2.5	0.4
* Nea Silata (irrigation)	7.6	3.1	0.5
Nea Triglia (drinking water)	0.9	1.8	0.6
* Nea Triglia (drinking water)	1.2	2.0	0.5
Nea Plagia (drinking water)	1.4	15.1	0.5
* Nea Plagia (drinking water)	0.9	16.9	0,5
Nea Plagia (drinking water)	1.3	1.2	0.4
* Nea Plagia (drinking water)	1.2	1.0	0.3

* Acidified water samples

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Further, it is noteworthy that non-acidified and acidified sets of water samples showed no significant differences in their values except of two samples of irrigation water from Sozopolis and Tenedos villages, which means that there was no particular element loss in non-acidified water samples in comparison with the acidified ones during the sample collection step until the analysis.

Conclusions

The results are in normal levels for all the three heavy metals for all kinds of water at these villages of municipality of Poligiros, Chalkidiki apart from geothermal water at Eleochoria village and in some cases for irrigation water at Nea Triglia and Tenedos villages for which the levels of arsenic were a little elevated. Geothermal water usually exhibits elevated levels of arsenic and its use as irrigation and/or drinking water has as a consequence the contamination of these kinds of water. So, the use of geothermal water as irrigation and/or drinking water should be prohibited.

The methods of Hydride Generation-Atomic Absorption Spectroscopy and Inductively Coupled Plasma-Mass Spectrometry which were used in the present work proved to have high accuracy and low detection limits for analysis of trace elements in water.

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