Penerbit Akademia Baru

Journal of Advanced Research Design

Journal homepage:
www.akademiabaru.com/ard.html
ISSN: 2289-7984

Microwave Plasma Atomic Emission Spectroscopy (MP-AES): Alternative Spectroscopy Method for Heavy Metals Analysis in Water

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ARTICLE INFO

Article history:
Received 29 March 2024
Received in revised form 17 July 2024
Accepted 25 July 2024
Available online 1 August 2024

Keywords:
MP-AES; heavy metals; lake water; tap water

ABSTRACT

Water is one of the necessities in our daily life, yet its pollution poses risks to human and ecological well-being. In this study, the presence of eight heavy metals in samples collected from two lakes and five tap water sources were investigated using microwave plasma atomic emission spectroscopy (MP-AES). Utilizing an alternative spectroscopy method, MP-AES able to achieve detection limits in the sub-parts per billion (ppb) level. Analysis of the water samples revealed that all eight metals were detected within a concentration range, from undetectable levels up to 0.93 mg/L. Moreover, the recovery on sample spike demonstrated an acceptable range of 107-114 %. These findings underscore the potential of MP-AES as a promising method for the analysis of heavy metals in water, offering valuable insights for water quality assessment and management.

1. Introduction

Water is important in life, as we are made up of 75 % of water. The presence of heavy metals in waters are caused by leaching from surrounding pipes [1], waste from factories [2] and contamination from agriculture [3]. Heavy metals are considered as trace elements when their presence is in ppb to less than 10 ppm ranges. High and unusual concentrations of toxic metals can become a problem in lakes or rivers as it contains a diverse biological organism [3]. In addition to that, heavy metals can enter the human body through water, air and food consumption [4]. Exposure to these toxic metals can cause toxic effects from subtle symptoms to major disorders [5].

Heavy metals are toxic, poisonous [6] and are non-biodegradable in nature [7]. Examples of heavy metals include lead, copper, zinc, nickel, chromium and cadmium. The World Health Organisation (WHO) has prepared a guideline for heavy metal limits in water and their effect upon exposure. Lead (Pb) exposure has been associated with a myriad of neurodevelopmental effects, increased mortality rates (primarily due to cardiovascular diseases), diminished renal function, hypertension, impaired

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https://doi.org/10.37934/ard.118.1.19

fertility and adverse pregnancy outcomes [8]. On the other hand, manganese (Mn) in water supplies causes a bad taste in beverages, and staining the utensils and cloths at levels greater than 0.1 mg/L. At concentration of 0.2 mg/L, pipes will develop a layer of Mn that may flake off as a black precipitate. The presence of iron (Fe) promotes the growth of "iron bacteria," causing the oxidation of ferrous ions (Fe^{2+}) to ferric ions (Fe^{3+}) and depositing a slimy coating on the pipe. At concentrations greater than 0.3 mg/L, Fe will stain laundry and plumbing fixtures, while at concentration below 0.3 mg/L, no discernible taste, though turbidity and colour may develop. Copper (Cu) may give a colour and an unpleasant bitter taste to water at levels above 5 mg/L. According to WHO, 2 mg/L is an acceptable Cu value for health-based guidelines. The main sources of aluminium (Al) in drinking water are naturally occurring aluminium and aluminium salts employed in water treatment as coagulants. At concentrations reaching 4 mg/L, zinc (Zn) imparts an undesirable astringent taste to water. Boiling water containing zinc concentrations exceeding 3-5 mg/L may result in an opalescent appearance and the formation of a greasy film [8].

Heavy metals possess detrimental effects even at a low level in the environment. Therefore, the effective and precise detection of heavy metals presence in the environment is a crucial requirement for pollution assessment and prevention since they serve as a potent tracer for observing the effects of human activities. The most common detection techniques used for heavy metals analysis are inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectroscopy (ICP-OES) and atomic absorption spectrometry [9–11]. All the analytical techniques provide good detection for trace metals [10] but require expensive gas for the operation [12].

Microwave plasma atomic emission spectroscopy (MP-AES) is another analytical technique used for element detection. This analytical technique can analyse multi-element, has a low operating cost, good detection power and provides fast detection [13]. A lot of studies have used MP-AES for metals or element detection from various samples such as ground water, soil, sediment, liqueurs, henna, fertiliser and honey [12,14–17]. MP-AES works like ICP-OES, but the main distinction between these two methods is the way to maintain the plasma. MP-AES operates under nitrogen-plasma supplied from compressed air and a nitrogen generator, which significantly reduces the operating cost. In this article, eight heavy metals – copper, zinc, aluminium, nickel, magnesium, manganese, lead and iron were analyzed from water samples (tap water, filtered water and lake water).

2. Methodology

2.1 Materials

Nitric acid (HNO_3, 69 %) was purchased from Merck, Germany. Working standards of the metals were prepared from metals salts: copper (II) nitrate trihydrate ($Cu(NO_3)_2.H_2O$), aluminium nitrate nonahydrate ($Al(NO_3)_3.9H_2O$), lead (II) nitrate ($Pb(NO_3)_2$), iron (II) nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$), magnesium nitrate hexahydrate ($Mg(NO_3)_2.6H_2O$), manganese (II) nitrate tetrahydrate ($Mn(NO_3)_2.4H_2O$), nickel (II) nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$) purchased from Merck (Germany), while zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$) was obtained from R&M Chemicals (UK). Ultra-pure water (18.2 MΩ.cm resistivity, Millipore Waters, USA) was used throughout the experiments and all chemicals were analytical grade.

2.2 Sample Collection and Preparation

Two lake water samples were collected from Pusat Marin, UPNM and Taman Tasik Cempaka, Bangi. Samples were collected at least three times per sampling point. Five tap water samples were

collected from the authors house (filtered and unfiltered), Bestari and Jauhari building, UPNM. Table 1 summarizes the collected water samples.

Table 1
List of water samples collected from different location

Type of water sample	Location	Label
Lake water	Pusat Marin, UPNM	PM
	Taman Tasik Cempaka, Bangi	TTC
Unfiltered tap water	Bangi	UF1
	Shah Alam	UF2
	Bestari Building	B
	Jauhari Building	J
Filtered tap water	Bangi	F

All water samples were prepared according to method 200.8 by the US Environmental Protection Agency (US EPA) [18]. The water was first filtered and centrifuged. Then, around 20 mL of the filtered water sample was pipetted into a 50 mL polypropylene centrifuge tube. The acid concentration of the filtered water sample was adjusted to approximately 1 % (v/v) HNO_3 solution by adding an appropriate volume of HNO_3. Each water sample was prepared triplicate and kept in the refrigerator before further analysis.

Two samples spike were prepared by spiking small amount of Zn stock standard solution into sample B and J. The recoveries of the sample spike were calculated using Eq. (1). Each sample spike was prepared and analyzed in triplicate.

$$\% \text{ Recovery} = \frac{\text{Spiked sample result-Unspiked sample result}}{\text{Known spike added concentration} \times \text{Dilution factor}} \times 100\% \quad (1)$$

2.3 Standard Solution Preparation

A 1000 mg/L stock standard solution for each metal salt was prepared by measuring out an appropriate weight of salt and diluted with ultra-pure water in 250 mL volumetric flask until the graduation mark. Preparation of 100 mg/L mixed standard solution was conducted by diluting stock standard solution with 1 % HNO_3. 25 mL from the stock solution of each metal was added into a single 250 mL volumetric flask and filled with 1 % HNO_3 until the graduation mark. Then, a series of calibration standards (0.1-2.0 mg/L) was prepared by diluting the 100 mg/L mix standard with 1 % HNO_3.

2.4 Instrumentation

Agilent 4210 MP-AES was used to determine the concentration of heavy metals. This instrument was equipped with a quartz torch, a glass concentric nebulizer and a cyclonic spray chamber. The analytical signal obtained from the analysis was evaluated using software MP Expert (Agilent Technologies). Operating parameters utilised in this study are presented in Table 2. The viewing position and nebulizer flow were optimized every time using the MP-AES.

Table 2
MP-AES operating parameter

Items	Units
Pump speed	15 rpm
Acquisition parameters	
Sample uptake time	20 sec
Stabilization time	10 sec
Read time	3 sec
Number of replicates	3
Analytes (wavelengths)	Ni (352.454 nm)
	Mg (285.213 nm)
	Cu (327.395 nm)
	Pb (405.781 nm)
	Zn (213.857 nm)
	Mn (403.076 nm)
	Al (396.152 nm)
	Fe (371.993 nm)

3. Results

3.1 Analytical Performance

The limit of detection (LOD) and limit of quantification (LOQ) were determined by analyzing different concentrations of eight heavy metals (Ni, Mg, Cu, Pb, Zn, Mn, Al and Fe) through MP-AES. For all eight heavy metals, the lowest concentration able to be detected and quantified by the MP-AES are in sub-ppb level, around 2.5 µg/L and 10 µ/L, respectively. These LOD and LOQ shows a similar result by [19] with sub-ppb limit.

External standard calibration using 1 % HNO_3 was employed to conduct linear regression analysis for each heavy metal. The parameters of each calibration curve, including slope, intercept and coefficient of determination (R^2), are depicted in Figure 1. For each of the examined heavy metals, good linearity between intensity and concentration was observed over the measured range ($R^2 \geq 0.99$).

3.2 Analysis of Heavy Metals in Water

Metals like cobalt (Co), copper (Cu), iron (Fe), magnesium (Mg), nickel (Ni), zinc (Zn) and manganese (Mn) are important nutrients that are needed for biochemical and physiological functions. If there is an inadequate supply of these nutrients it can result in deficiency diseases or syndromes. In plants and animals these micronutrients have roles in oxidation-reduction reactions and are important components in few key enzymes [20]. However, these metals are considered heavy metals that are five times denser than water and have a high atomic weight. Heavy metals turn poisonous when they build up in soft tissues without being metabolised by the body. Both natural and anthropogenic processes can release heavy metals, which then end up in various environmental compartments like soil, water and air [21].

Table 3 summarizes the concentration of eight metals detected from seven water samples. The presence of excessive levels of these heavy metals in water may serve as an indicator of the severity of water pollution. In this study, seven water samples were analyzed for the presence of eight heavy metals. The findings from the quantitative analysis conducted using the MP-AES method revealed that only certain heavy metals were detected. Among all water samples, only the filtered water sample (F) did not contain most of the heavy metals analyzed except for Mg. This is expected due to the filtration capable to eliminate almost all the unwanted metals [22].

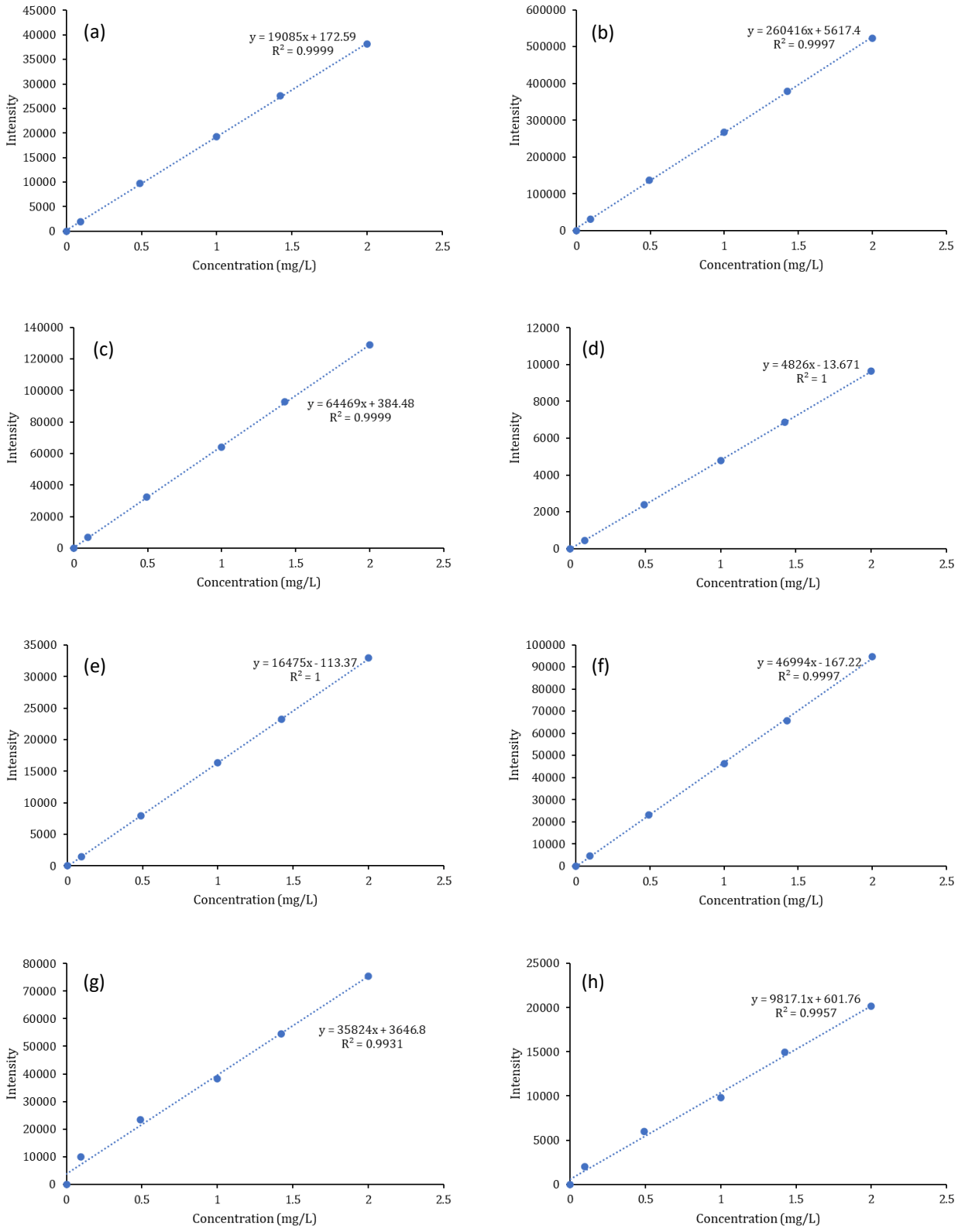

Fig. 1. Calibration curve of the examined heavy metals: (a) Ni, (b) Mg, (c) Cu, (d) Pb, (e) Zn, (f) Mn, (g) Al and (h) Fe

Table 3
Concentration of heavy metals detected by MP-AES in each water samples

Label	Concentration (mg/L)							
	Ni	Mg	Cu	Pb	Zn	Mn	Al	Fe
F	N.D	0.88	N.D	N.D	N.D	N.D	N.D	N.D
UF1	N.D	0.85	N.D	N.D	0.02	N.D	N.D	N.D
UF2	N.D	0.79	N.D	N.D	0.15	0.01	N.D	N.D
B	N.D	0.66	N.D	N.D	0.41	0.01	N.D	0.08
J	N.D	0.62	N.D	0.02	0.01	0.03	N.D	0.03
PM	N.D	0.68	N.D	N.D	0.01	0.04	N.D	0.22
TTC	N.D	1.47	N.D	N.D	0.02	0.05	0.02	0.93

Note: N.D = not detected

The findings of heavy metals level were subsequently compared to the guidelines established by the WHO, the European Union and the National Water Quality Standard for Malaysia (Table 4). Water quality guidelines serve as criteria or declarations designed to protect aquatic ecosystems and various human water-related activities and interests [23]. These guidelines play an important role in protecting water resources, guiding assessments of water quality, facilitating resource management choices, establishing water quality objectives, reporting on water quality status and encouraging responsible water management practices [24].

The detected concentrations varied between 0.01 and 1.47 mg/L. It is worth mentioning that magnesium (Mg) was the predominant heavy metal found in all water samples, with concentrations ranging from 0.62 to 1.47 mg/L. Magnesium (Mg) is naturally present in water and is the most prevalent alkali metal in the environment [25]. However, neither the WHO nor the National Water Quality Standards and Water Quality Index of Malaysia has established a guideline limit for magnesium concentration in water [8,26]. Despite magnesium being the most abundant metal in all water samples, its concentration is not harmful to humans as long as dietary intake does not exceed 400 mg [27]. Therefore, its presence does not pose a risk to human health.

Nickel (Ni) and copper (Cu) were not present in any of the samples, while lead (Pb) and aluminum (Al) were also absent in all samples except for water sample J and TTC, respectively. For water sample J, the concentration of Pb (0.02 mg/L) exceeded the WHO standard but still acceptable by the European Union standard and National Water Quality Standard for Malaysia. The possible source of dissolved lead present in tap water sample J could be due to leaching from lead pipes, galvanized steel pipes and PVC pipes that are plasticized with lead [28,29]. Although Al is detected in water sample TTC, the amount of Al (0.02 mg/L) in did not exceed the permissible level stated by WHO, European Union and DOE Malaysia. The similar pattern also seen in Manganese detected in five water samples. However, the concentration detected (0.01-0.05 mg/L) not exceeding all the regulations, hence it is considered not harmful to human or aquatic live.

Table 4
Guideline for water quality from different regulations

Regulation	Concentration guideline (mg/L)								Reference
	Ni	Mg	Cu	Pb	Zn	Mn	Al	Fe	
WHO	0.07	-	2.00	0.01	0.01	0.40	0.90	0.30	[8]
European Union	0.02	-	2.00	0.05	-	0.05	0.20	0.20	[31]
National Water Quality Standard for Malaysia	0.05	-	0.02	0.05	5.00	0.10	-	1.00	[32]

In addition to that, out of seven water samples, six of them detected Zn with concentrations ranging from 0.01 to 0.41 mg/L. According to Noulas *et al.,* [30] Zn found in water sample could come from various sources including mine drainage, industrial and municipal waste, urban runoff and primarily through the erosion of soil particles containing zinc [30]. Three of tap water contained Zn more than the permissible level stated by WHO, but still acceptable based on the National Water Quality Standard for Malaysia.

Iron (Fe) can be found in natural fresh water, but at a low concentration ranging from 0.5 to 50 mg/L [6,8]. Fe is an essential nutrient in humans, especially in the iron (II) oxidation state. Estimation of the minimum daily requirement for Fe ranging from 10 to 50 mg/day, depending on age, gender, physiological status and Fe bioavailability [8]. According to WHO, the acceptable threshold for Fe in drinking water is 0.3 mg/L. However, increased levels of Fe in water can be attributed to the disposal of domestic and industrial waste into water bodies [22]. In this study, high concentrations of Fe are detected in two lake water PM and TTC, with concentration of 0.22 and 0.93 mg/L, respectively. The presence of Fe in lake water could come from the soil or sediment containing Fe or rainwater. Water sample TTC exceeded the permissible limit regulated by the WHO and European Union. Although the level of Fe in TTC water sample still acceptable by National Water Quality Standard for Malaysia, but precaution need to be taken as it almost reaches the limit as high concentration of Fe can adversely affect the quality water [33].

3.3 Analysis of Spiking Water Sample

The accuracy of the method analysis for real sample application was assessed by spiking water sample B and J with a known concentration of Zn standard solution (around 0.47-0.64 mg/L). The percentage recovery for spike water sample B and J is displayed in Table 5, and it varies between 107 to 114 %, which falls within the acceptable range and shows the validity of the optimized MP-AES method.

Table 5
Recovery on spiked water sample

Water sample	Recovery ± SD (%)
B	107 ± 5
J	114 ± 3

4. Conclusions

In this study, MP-AES was used to analyze eight heavy metals (Ni, Mg, Cu, Pb, Zn, Mn, Al and Fe) in seven water samples; two lake waters and five tap waters. MP-AES offers fast sequential determination of multiple elements with sub-ppb detection limit. Furthermore, since it extracted nitrogen extracted from air using a nitrogen generator, thus this approach is safer and incurs low operational cost. All water samples with heavy metals concentration recorded are still acceptable by the National Water Quality Standard that was set by the Department of Environment (DOE), Malaysia. With this study, it can provide the data for future guidelines, particularly for the evaluation of lake and tap water using MP-AES method.

Acknowledgement
This project was financially supported by the National Defence University of Malaysia through the *Geran Penyelidikan Jangka Pendek (GPJP)* Scheme (UPNM/2023/GPJP/STG/1). The authors would like to thank National Defence University of Malaysia for providing the facilities for conducting the experiment and University Technology MARA for internship programme.

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