

REVIEW ARTICLE

Bibliometric analysis (VOSviewer): Analytical chemistry techniques for heavy metals and lead determination in Iraq: A review

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ABSTRACT

This review provides a comprehensive overview of heavy metal and lead analysis in environmental, drug, cosmetic, and food pollution samples, focusing on research conducted in Iraq. The explore of the co-occurrence of heavy metals and analytical chemistry techniques in published studies over the last ten years was achieved by using bibliometric analysis (VOS viewer) based on database from the Scopus. For determining heavy metal concentrations in different environmental samples, various analytical chemistry techniques were assesses in this review such as Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Atomic Absorption Spectrometry (AAS), and X-ray fluorescence (XRF). This study confirms the importance of development of efficient, selective, and sensitive techniques for heavy metal detection in Iraq to reduce their harmful impacts on human health and the environment.

Keywords: VOSviewer; heavy metals; analytical chemistry; bibliometric analysis; Iraq

ARTICLE INFO

Received: 30 March 2025

Accepted: 25 April 2025

Available online: 07 May 2025

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1. Introduction

Heavy metals are metallic elements with a relatively high atomic weight and density compared to other substances in the periodic table^[1]. Heavy metals are found in soils, water, organism bodies, and all our biosphere^[2]. Most of these metals, such as (As, Cd, Pb, Ni, and Hg), pose a significant health risk, even at low concentrations or when exposure exceeds permissible daily limits^[3]. The negative health effects for human come from when elements are toxic. These toxic elements can bioaccumulate in human body, causing disorder with physiological functions or illness^[4].

The risk control of elemental impurities in drug products is describes by International Council for Harmonization (ICH) Q3D guidelines. The risk assessment of elemental impurities includes manufacturing equipment, excipients, drug substance, utilities, and container closure system^[5]. PDE is the permitted daily exposure limits of elemental impurities for pharmaceutical product^[6]. The maximum elemental impurities consumption of oral drug dosage is 10 g day per day. ICHQ3D guideline classifies of elements to the four classes based on their potential to appear as contaminants in pharmaceutical products and their toxicity, they are^[7-9]:

Class 1: elements of significant human toxicity (As, Cd, Pb, and Hg). These elements have no therapeutic benefit and found in drugs from raw materials like mined excipients.

Class 2: Elements of moderate human toxicity and divided into two subclasses: Class 2A includes (Ni, Co, and V) which are high presence element as impurities in drugs. Class 2B includes (Au, Ag, Ti, Se, Ru, Rh, Pt, Pd, Os, and Ir) which are low probability of presence element as impurities in drugs.

Class 3: Elements of low human toxicity, when administered via oral route, intramuscular, and intravenous route (Li, Cr, Sn, Cu, Mo, Ba, and Sb). The PDE limits exceeding of 500 µg/day.

Moreover, some of elements not classified in the above classes due to their low inherent toxicity include: Al, Zn, W, Ca, Na, Mn, K, Fe, B, and Mg. The classification of elements and their PDE limits for oral consumption according to the ICH Q3D guidelines are shown in **Table 1**.

Lead (Pb) a particularly hazardous heavy metal within this category. Lead, known as "*plumbum*" in Latin, and sometimes referred to as liquid silver, has an atomic number of 82 on the periodic table. Its presence in drug products can originate from commonly used minerals, raw materials, excipients, or metal reagents and catalysts employed in the synthesis of active pharmaceutical ingredients (APIs)^[10,11].

The network data obtained from scientific literature databases (such as Scopus, Web of Science, and PubMed) can be used in VOSviewer software for bibliometric network analysis. It is used to create maps that provide a comprehensive understanding of the analytical chemistry techniques used for the determination of heavy metals in different complex samples and that show the linkage between scientific publications focusing on pollutants in soil, rivers, drugs, blood, and keywords of heavy metals through co-occurrence bibliographic analysis.

This review aims to provide an overview of heavy metal and lead analysis, especially within the context of Iraq using bibliometric analysis (VOS viewer) and to assess the analytical chemistry techniques such as AAS, ICP-OES, and ICP-MS used for determination of heavy metals.

Table 1. Element toxicity classification according to ICH Q3D guidelines.

Element	Oral PDE (µg/day)	Class (Toxicity)
Cd	5	1 strong
Pb	5	
As	15	
Hg	30	
Co	50	
V	100	2A (Moderate)
Ni	200	
Ti	8	
Au	100	
Pd	100	
Ir	100	2B (Moderate)
Os	100	
Rh	100	
Ru	100	
Pt	100	
Se	150	3 (Moderate)
Ag	150	
Li	550	
Sb	1200	
Ba	1400	
Mo	3000	
Cu	3000	
Sn	6000	
Cr	11000	

2. Methodology

This bibliometric study, conducted from 2015 to 2025, used the Scopus database to analyze the application of analytical chemistry techniques for the determination of heavy metals in Iraq. The analysis was structured depend on the following stages:

1. We focused on the co-occurrence network of (A) heavy metals and (B) analytical chemistry techniques used for their analysis in the world (**Figure 1**).The search strategy employed selected keywords from Scopus database, including ("heavy metals" AND "analytical chemistry techniques").
2. We studied the co-occurrence of analytical chemistry techniques specifically used for the analysis of lead metal in the world (**Figure 2**). The search used keywords including ("heavy metals" AND "analytical chemistry techniques" AND "lead").
3. We studied the global application of analytical chemistry techniques for the analysis of lead metal in pharmaceutical formulations over the last ten years (**Figure 3**).The search used keywords including ("heavy metals" AND "analytical chemistry techniques" AND "lead" AND "pharmaceutical formulations").
4. We focused on the analytical chemistry techniques used for the analysis of heavy metals in Iraq (**Figures 4, 5, and 6**). The search used keywords including ("heavy metals" AND "analytical chemistry techniques" AND "Iraq").

2.1. Analytical chemistry techniques for analysis of heavy metals

In this study, we utilized the VOS viewer software tool to construct bibliometric networks for heavy metals and the analytical chemistry techniques used for their analysis^[12-14]. **Figure 1A** shows the co-occurrence network of heavy metals found in research publications over the past ten years based on database from the Scopus. The big size of node corresponds to the more prevalent the heavy metal and the lines connecting the nodes represent the co-occurrence of these heavy metals. Moreover, **Figure 1B** shows the co-occurrence network of analytical chemistry techniques used for heavy metals analysis found in research publications over the past ten years. The big size of node corresponds to the more prevalent analytical chemistry techniques, such as Atomic Absorption Spectrometry (AAS) and Mass Spectrometry. The links between nodes indicate which techniques are frequently mentioned together, like AAS technique is strongly linked to spectrophotometry, and spectrometry techniques, indicating that these techniques are often used in conjunction with each other^[15-17].

complex matrices (water, soil, blood, urine, and samples tissues)^[18-21]. Spectroscopic techniques (scanning electron microscopy, X ray diffraction, nuclear magnetic resonance SPE) are probably used for identifying and quantifying lead at trace levels^[22,23]. Electrochemical methods (electrodes and electrochemical techniques) are likely used for determining lead concentrations based on its electrochemical properties^[24,25]. Keywords related to the microfluidic analytical method appeared less frequently for lead analysis compared to terms associated with other techniques^[26].

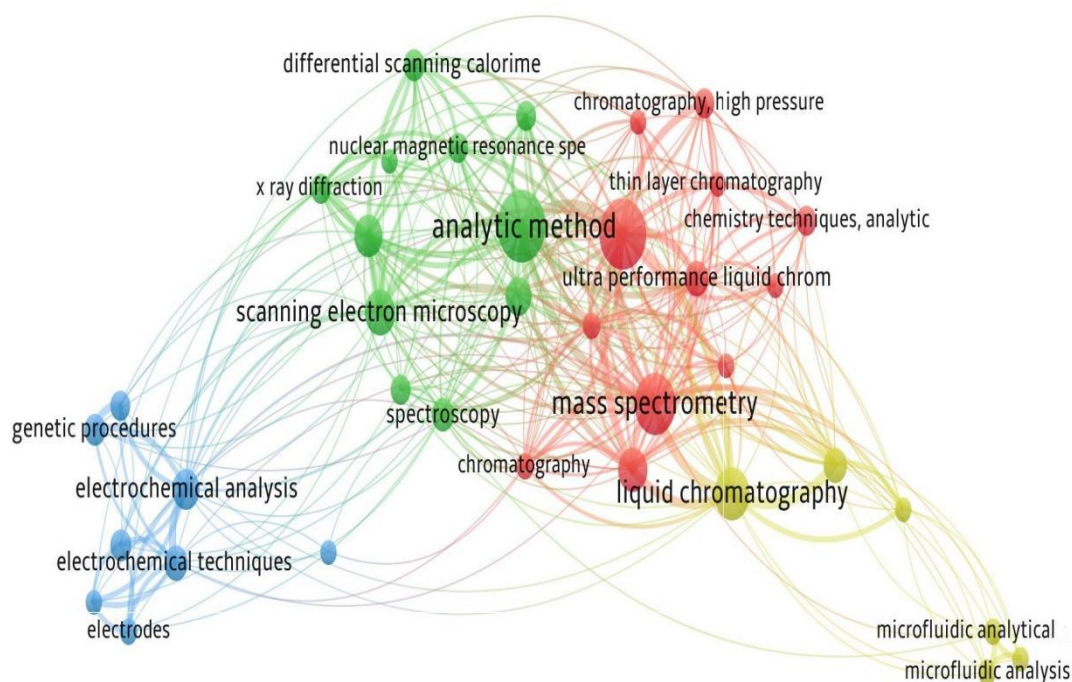


Figure 2. The co-occurrence network of analytical chemistry techniques used for analysis Lead metal.

Chromatographic techniques (essentially HPLC) are the most analytical tools used for lead analysis in pharmaceutical formulations over the past ten years as shown in **Figure 3**. This is due to their ability to separate and quantify lead species in complex matrices (such as blood, urine, serum, tissue, and complex pharmaceutical formulations), high sensitivity, and selectivity^[27,28]. Spectroscopic techniques (such as FTIR, SEM, and XRD) play a significant role, but less than chromatographic method^[29,30]. Electrochemical and biosensing techniques are less dominant, but they are also used for lead detection and quantification in pharmaceutical formulations^[31,32].

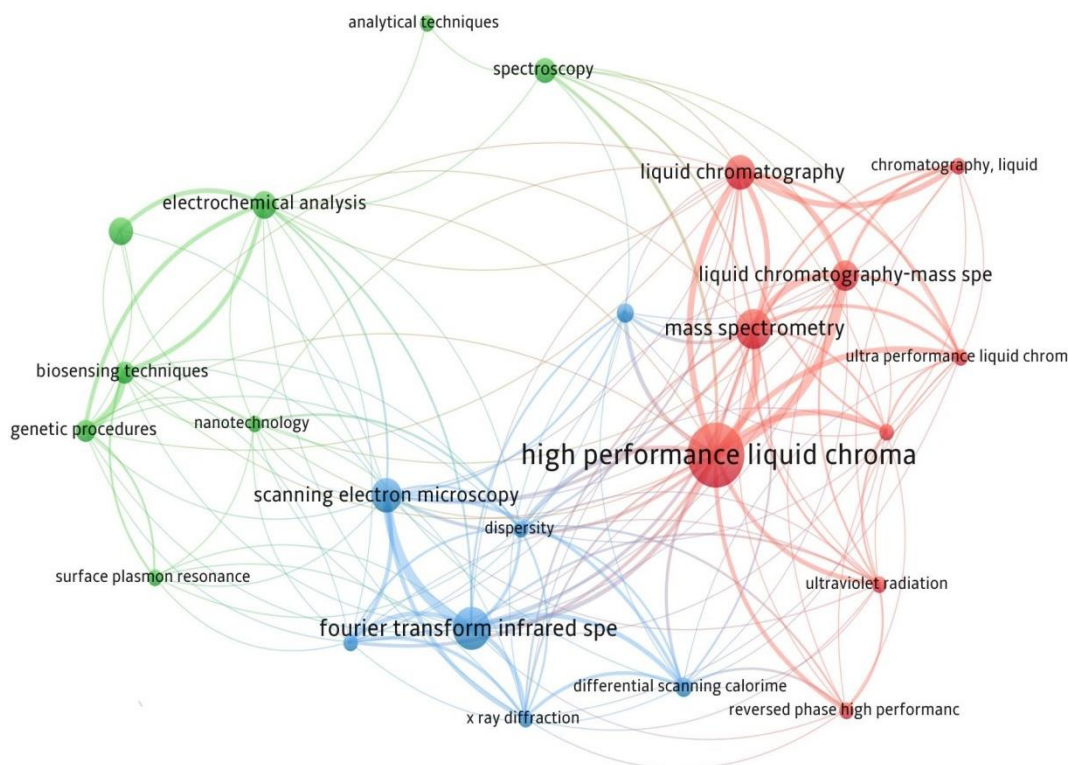


Figure 3. The co-occurrence network of analytical chemistry techniques used for analysis Lead metal in pharmaceutical formulations over the last ten years.

2.3. Analytical chemistry techniques for analysis of heavy metals in Iraq

In the past ten years, only two studies, utilizing AAS, analyzed zinc, iron, and lead in medicinal plant-derived pharmaceutical samples (solid and liquid) and determination (Ni, Co, Fe, Pb, Cd and Cr) by using AAS in Aspirin tablets available in Iraqi markets^[63,59]. Therefore, this study will be focuses on the analytical chemistry techniques used for heavy metal analysis in different samples in Iraq. **Figure 4** shown the co-occurrence network related to the analysis of heavy metals within research publications from Iraq over the past ten years. Nodes such as Cd, Hg, Cr, Ni, Cu, Ar, and Pb, indicates these heavy metals are the most frequently studied in Iraq^[33, 34].

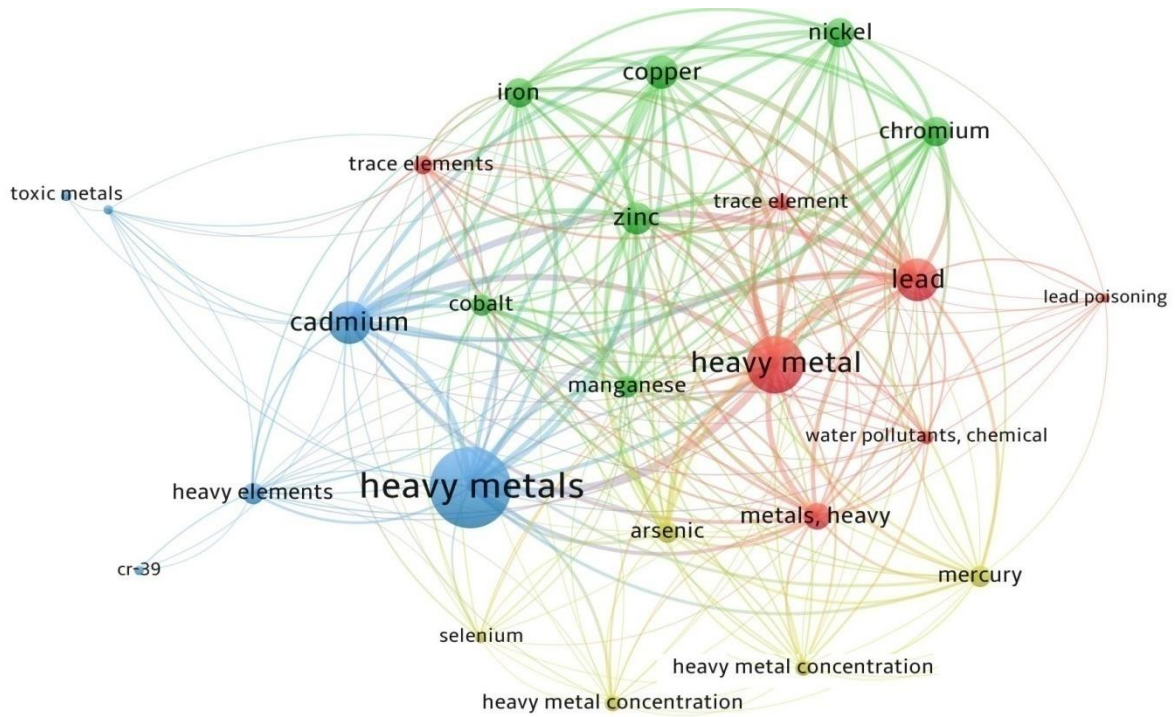


Figure 4. Heavy metals analysis in Iraq over the last ten years.

Figure 5 demonstrates the dominance of water pollution in Iraq, particularly concerning heavy metals. The figure highlights that pollution in rivers, drinking water, lakes, groundwater, and wastewater, including the Tigris and Euphrates rivers, is a major concern. Additionally, soil pollution, dust, air pollution, and specific sources like animals, crude oil, and the petroleum industry are also mentioned as contributing to pollution^[35-39].

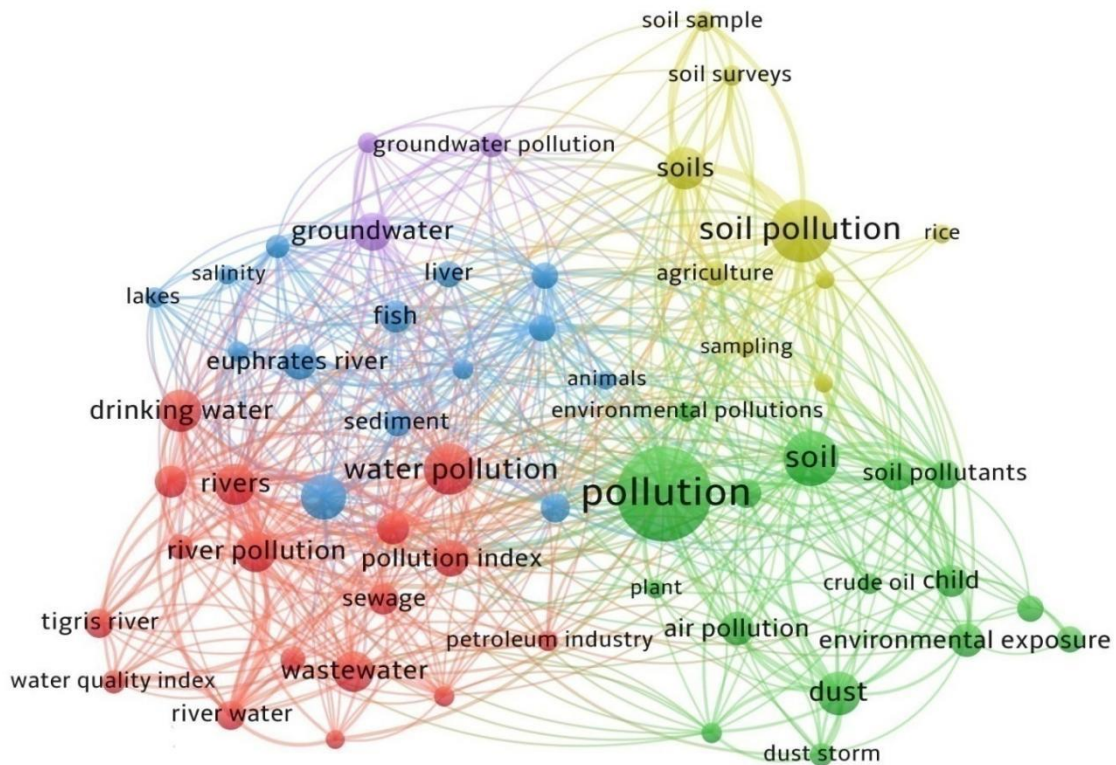


Figure 5. Pollutant samples with heavy metals in Iraq over the last ten years.

The largest node size indicated that the Atomic Absorption Spectrometry (AAS) and Flame Atomic Absorption Spectrometry (FAAS) are the most analytical tools used for determination of heavy metals in Iraqi land space over the past ten years as shown in **Figure 6**. This could be due to factors like its cost-effectiveness, accessibility, and suitability for analyzing heavy metals prevalent in the Iraqi region. This makes it a more accessible option for many laboratories in Iraq, and it is well-suited for the determination of many heavy metals commonly found as pollutants in soil, rivers, drugs, blood, and other matrices pollutant in Iraq. The other techniques (such as X-ray Diffraction, ICP, and X-ray diffraction) play a significant role, but less than AAS and FAAS methods. Finally, Scanning electron microscopy (SEM) was used to investigate the surface morphology of pollutant materials. **Table 2** shows a summary of analytical chemistry techniques such as AAS, XRF, and ICP-MS used to determine heavy metals in various samples from different regions of Iraq.

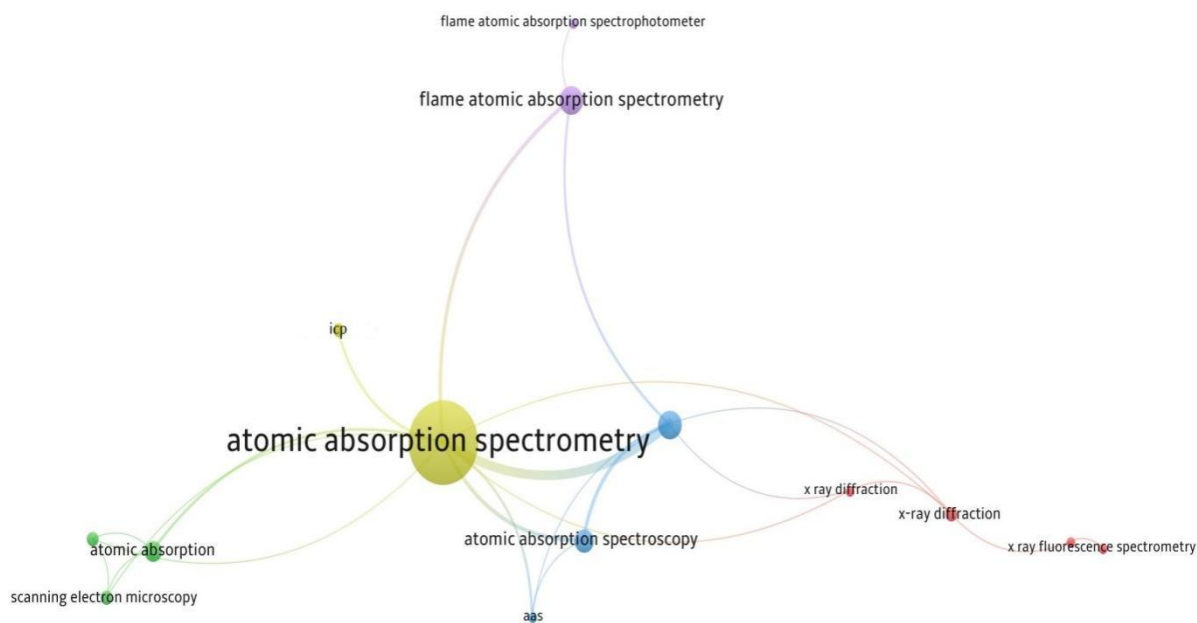


Figure 6. The co-occurrence of analytical chemistry techniques used for determination heavy metals in Iraq over the last 10 years.

Table 2. Analytical chemistry techniques used to determination of heavy metals in Iraq.

Analytical Chemistry Techniques	Metal	Region	Sample	Year	Ref.
Flame Atomic Absorption Spectrophotometer (FAAS)	lead, cadmium, copper and iron	Shatt Al-Arab	water	2023	34
X-ray fluorescence (XRF)	lead , aluminium , chromium, nickel , and arsenic	Sulaymaniyah Governorate	concrete blocks	2025	35
Inductively coupled plasma mass spectrometry (ICP-MS)	As, Cd, Co, Cr, Ni, Pb	Al-Qayarah city	soil samples	2024	36
Flame Atomic Absorption Spectroscopy (FAAS)	Zn, Co, Cu, Pb, Cd, Cr, Fe, and Ni	Anbar Province	soil of oil installations	2024	37
Flame atomic absorption spectroscopy (FAAS)	zinc, copper, nickel, lead, and cadmium	Karbala	tap water samples	2024	38
Atomic Absorption Spectrometry (AAS)	lead, zinc, cadmium, nickel, copper, and chromium	Nine cities in Iraq (Basra, Najaf , Kirkuk , Baghdad , Hillah, Diwaniyah, Samawah, Kut, Amarah)	dust storms	2023	39
Flame atomic absorption spectroscopy (FAAS)	Cadmium, lead, Copper, Zinc, and Iron	Al-Hawizeh Marshes, Misan Province	liver tissues of three migratory bird (Anas platyrhynchos, A. crecca, and A. acuta)	2023	40

Analytical Chemistry Techniques	Metal	Region	Sample	Year	Ref.
Atomic Absorption Spectrometry (AAS)	Cu, Hg, Zn, Cd, Pp	Al-Diwaniyah Governorate	blood samples	2024	41
Atomic Absorption Spectrometry (AAS)	Iron, Copper, Manganese, Cobalt, Nickel, Cadmium and Copper	Baiji in Salah al-Din Governorate	Tigris River, Groundwater, and sewage drain	2022	42
Inductively coupled plasma optical emission spectroscopy	Fe, Mn, Ni, Cu, Zn, Hg, Al, Ba, Se, and As	Diyala-Sirwan river	wastewater discharge on water river	2021	43
Atomic Absorption Spectrometry (AAS)	lead	Kalak city, Erbil	Cowpea seeds and soil	2015	44
Atomic Absorption Spectrometry (AAS)	Ni, Zn, Co, Cr, and Pb	Al-Najaf refinery	soil	2025	45
Atomic Absorption Spectrometry (AAS)	Zn, Pb, Cd, Cu, Co, and Fe	Great Musaiyab, Babylon	water samples	2024	46
Atomic Absorption Spectrometry (AAS)	Nickel, Cadmium, and Lead in the liver and small intestine tissues of Marbled Teal.	Al-Dalmaj marsh	Liver and small intestine tissues of Marbled Teal.	2024	47
Atomic Absorption Spectrometry (AAS)	Mercury and Lead	Baghdad	Blood samples	2024	48
Atomic Absorption Spectrometry (AAS)	cadmium, lead, zinc, and mercury	Al-Hindiyah District, Al-Hindiyah District, and Al-Kifl District	Euphrates River	2024	49
Atomic Absorption Spectrometry (AAS)	Fe, Mn, Cd, Co, As, Hg, Pb, and Se	the vicinity brought on by the Erbil Steel Factory	Rainfall	2024	50
Atomic Absorption Spectrometry (AAS)	Ni, Cu, Cd, Pb, and Co	Maysan Refinery, Al-Masharah	Groundwater	2024	51
Flame atomic absorption spectroscopy (FAAS)	Cu, Zn, Pd, Cd, Mn, Mg, Ni, and Fe	Baghdad	Blood samples of fast-food workers	2024	52
Inductively Coupled Plasma Emission Spectrometry (ICP-AES)	Lead	Diyala River, AL Rustamiya, Karbala, Babylon, and AL-Rashdiya	Cyprus papyrus, Tigris and Euphrates rivers	2024	53
Energy dispersive X-ray fluorescence (EDXRF)	Cd, Pb, Ni, Cu, and Zn	Kurdistan	Milk powder	2025	54
X-ray fluorescence (XRF)	Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Cd, Sb, Hg, and Pb	Left Side of Mosul	Street Dust	2024	55
Flame Atomic Absorption Spectroscopy (FAAS)	Fe, Pb, Cu, Cr, and Zn	Basra	Water and blood samples	2020	56
X-ray fluorescence (XRF)	Cr, Co, Zn, Ni, Cu, Pb, Cd, and Zr	Kut	Tigris River sediments	2019	57
X-ray fluorescence (XRF)	Cr, Ni, Pb, Cd, and As	Okab industry- west of Mosul City and Karama Industry-East of Mosul City	Soil	2020	58
Atomic Absorption Spectrometry (AAS)	Nickel, Cobalt, Iron, Lead, Cadmium and Chromium	Iraqi markets	Aspirin tablets	2018	59
X-ray fluorescence (XRF)	Ti, Cr, Sr, Fe, Zn, Cu and Pb	Baghdad and ThiQar	Soil	2023	60
Inductively Coupled Plasma Emission Spectrometry (ICP-AES)	lead, mercury, and cadmium	Iraqi markets	Natural pharmaceutical products (NPPs)	2022	61
Atomic Absorption Spectrometry (AAS)	arsenic, mercury and cadmium	Iraqi markets	Rice types	2018	62
Atomic Absorption Spectrometry (AAS)	Zinc, iron, and lead	Iraqi markets	Drugs samples from medicinal plants (solid and liquid)	2023	63
Atomic Absorption Spectrometry (AAS)	Aluminum, Copper, Iron, Zinc,	Samaraa Drug Industry	Samaraa Drug Industry Water	2022	64

Analytical Chemistry Techniques	Metal	Region	Sample	Year	Ref.
	Manganese, and Chromium				
Atomic Absorption Spectrometry (AAS)	Cu, Fe, Zn, Pb,	drugs industry of Samarra	piped water	2021	65
X-ray fluorescence (XRF)	Fe, Co, Ni, Cu, Zn and Pb	Diwaniyah city-Qadisiyah Governorate areas near the fuel stations in Karkh	Road-side dust samples	2018	66
Atomic Absorption Spectrometry (AAS)	Cd, Ni, and Pb	District of Baghdad and sides of the highway between Ramadi and Rutba	Roadside dust	2015	67
Flame Atomic Absorption Spectroscopy (FAAS)	Cu, Ni, Cr, Pb, and Cd	Kurdistan	Soil and legume seeds samples	2016	68
Flame Atomic Absorption Spectroscopy (FAAS)	Pb	Sulaimani	lipsticks	2015	69
Atomic Absorption Spectrophotometer (AAS)	Pb, Cd and Cu	Naseriya markets	Medicinal plants	2019	70
Atomic Absorption Spectrophotometer (AAS) and Inductively coupled plasma mass spectrometry (ICP-MS)	As, Hg, Cd, and Pb	river and marine environmental of Basra	sediment samples	2019	71
X-ray fluorescence (XRF)	As, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Th, U, V, and Zn	Abu-Ghraib Land, Baghdad	plant samples of vegetables(carrot, onion, eggplant, cucumber, and okra)	2024	72
X-ray fluorescence (XRF)	Se, Sb, Te, Hg, Cd, Bi, U, Ni, Mo and As	Baiji and Salah Al-Din General Hospitals in Salah Al-Din	Dust Samples	2024	73
X-ray fluorescence (XRF)	Fe, Co, Ni, Cu, Zn and Pb	East of Baghdad	Streets dust	2021	74
Inductively coupled plasma mass spectrometry (ICP-MS)	Cu, Cr , Ni , Pb, As, Co, and Cd	Kirkuk City	Khasa River	2020	75
Inductively Coupled Plasma Emission Spectrometry (ICP-AES)	Arsenic, Cadmium and Lead	Al Delmaj Marshes	Water,Sediments and Fish	2019	76
Inductively coupled plasma mass spectrometry (ICP-MS)	Cd, Pb, Hg, Cr, Cu and Zn	Basra	blood, serum and hair of breast cancer patients	2021	77

Table 2. (Continued)

3. Conclusion

The bibliometric analysis (VOSviewer) based on data from Scopus revealed that Cd, Hg, Cr, Ni, Cu, As, and Pb are the most frequently investigated heavy metal contaminants in the Iraqi region, and that an appearing emergency issue in water pollution includes the Tigris and Euphrates rivers, groundwater, lakes, wastewater, and drinking water. The VOSviewer revealed that analytical chemistry techniques used for heavy metal determination in Iraq like AAS and FAAS. While other techniques are used less prevalent in Iraq such as ICP, X-ray Diffraction and X-ray fluorescence (XRF). The analyses of pharmaceutical samples are limited number of studies (just one) in Iraq, which is led to increase the research into pharmaceutical samples. Finally, the evaluation of various environmental samples need to more comprehensive research of heavy metal contamination in Iraq.

Declaration of competing interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Data availability

Data will be made available on request.

Acknowledgment

We thank Mustansiriyah University, College of Science, Department of Chemistry for their help.

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