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Review

Analytical techniques for estimation of heavy metals in soil ecosystem: A tabulated review

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ABSTRACT

Soil, an important environmental medium, is exposed to a number of pollutants including toxic heavy metals by various natural and anthropogenic activities. Consequently heavy metal contaminated soil has the potential to pose severe health risks and hazards to humans as well as other living creatures of the ecosystem through various routes of exposure such as direct ingestion, contaminated drinking ground water, food crops, contact with contaminated soil and through food chain. Therefore, it is mandatory to explore various techniques that could efficiently determine the occurrence of heavy metals in soil. A number of methods have been developed by several regulatory agencies and private laboratories and are applied routinely for the quantification and monitoring of soil matrices. The present review is an initiative to summarize the work on pollution levels of soil ecosystem and thus pertains to various extraction and quantification procedures used worldwide to analyze heavy metals in soil.

Contents

1. Introduction

Over the past decades, industrialization and mining activities led to emergence and dispersal of various pollutants in ecosystems. Among different contaminants, heavy metals were categorized as the most hazardous class of anthropogenic environmental

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pollutants due to their persistent nature and higher toxicity [1]. Furthermore, some heavy metals can enter the food chain thereby posing threat to the life of various biological systems at different trophic levels [2]. Therefore, human exposure to toxic metals via inhalation, food chain or dermal contact requires considerable attention [3]. It is well established that all metals including the micronutrient elements can turn out to be noxious at higher concentrations. The most important anthropogenic source of heavy metals for soils include agrochemicals such as pesticides and fertilizers, sewage from domestic sources, effluents from industrial activities as well as urban and vehicular emissions [4]. As soil is one of the most important sink of heavy metals, the emphasis has been given by many researchers to explore various techniques for analysis of heavy metals. There are many established analytical techniques, such as inductively coupled plasma optical emission spectroscopy (ICP-OES), inductively coupled atomic emission spectroscopy (ICP-AES), inductively coupled mass spectroscopy (ICP-MS), atomic fluorescence spectrometry (AFS), X-ray fluorescence spectrometry (XRF), neutron activation analysis (NAA), d.c. argon plasma multielement atomic emission spectrometry (DCP-MAES), scanning electron microscopy with energy dispersive X-ray and atomic absorption spectrometry (AAS) for the purpose of quantification and are considered to be efficient with good detection limits [5–8]. The present study focuses on the use of some of the widely used analytical techniques for estimation of heavy metal contents in different soil samples. Supplementary Table S1 summarizes literature on different analytical techniques along with digestion mixture used for estimation of heavy metals in soil ecosystem.

2. Analysis of heavy metals in soil ecosystem

The literature survey has revealed that atomic absorption spectrophotometry has been the most widely used technique for analysis of heavy metal content in soil matrix. A brief introduction of different techniques is given below.

2.1. Inductively coupled plasma optical emission spectroscopy (ICP-OES)

Inductively coupled plasma optical emission spectroscopy is demonstrated to be one of the most widely used techniques for estimation of heavy metal content in soils samples [9]. This technique has the feasibility to analyze the metals in traces. Though this technique was earlier considered as non-economical as compared to other techniques and had its limitations but the recent technological developments in ICP-OES has made it one of the important tools in field of heavy metal pollution analysis [10,11]. The major advantage of this technique is the increasing demand of performing simultaneous multielement determination at the nanogram/milliliter level for many trace constituents and limited volume of samples especially the biological samples. It was reported that even the most widely accepted technique like atomic absorption spectroscopy also does not meet these requirements [12].

Scott et al. [13] described the use of ICP-OES for trace determination of different metallic elements. They mentioned that detection limits were in range 0.1–10 ng/ml for most of the elements studied. The use of inductively coupled plasma as excitation source for trace metal estimations was first investigated by Greenfield and his associates in the year 1965. Since then, many authors demonstrated the feasibility of this technique in emission spectrometry [14–29]. Various studies have been reported on the use of ICP-OES for the determination of trace elements in soils from different regions of the world [30–53].

2.2. Inductively coupled atomic emission spectroscopy (ICP-AES)

ICP-AES spectroscopy was introduced for rock sample analysis by Govindaraju and Mevelle [54]. The use of AES for rock analysis was performed over the routinely used techniques like atomic absorption spectrometry and X-ray fluorescence spectrometry due to the fact that the instrument was capable of determining several elements simultaneously. Knasmuller et al. [55] also studied heavy metals *viz.* Cu, Ni, Cr, Cd, Hg, Sb and As in soils contaminated with emissions of smelters of Arnoldstein, Brixlegg, Slovakian (eastern Austria), Saualpe (central Austria) and Alps Carinthia region. Kim et al. [56] reported high contents of heavy metals from agricultural (paddy grown), farmland soils and dressing plant soil in the vicinity of Duckum Au–Ag mine, Duckum (Korea) using this technique. Li et al. [57] also reported the use of this technique for analyzing soils from urban areas (residential, commercial and industrial estates) and hotspots (volcanic regions) of Kowloon Peninsula (Hong Kong) for Cd, Co, Cr, Cu, Ni, Pb and Zn estimation. Rodriguez et al. [58] used ICP-AES technique for estimation of Pb, Zn, Cu and Cd in Arable and Pasture soils (polluted soils) in the vicinity of Pb–Zn mine area in Ciudad Real (Spain). Similarly several workers throughout the world have used Inductively coupled atomic emission spectrometer for estimating the contents of heavy metals in contaminated soil mixtures from different regions [59–82].

2.3. Inductively coupled mass spectroscopy (ICP-MS)

The multielemental ultratrace detection capability of inductively coupled plasma mass spectrometry (ICP-MS) makes it the most powerful technique [83]. Speciation analysis using inductively coupled plasma mass spectrometry (ICPMS) has become common practice in recent years for ultra-sensitive detection of trace metal- and metalloid-containing compounds [84]. It was prominent from survey of literature that this instrument has been used for industrial analysis of metals, chemicals and advanced synthesized materials [85,86], for environmental analysis [87–89] and clinical and biological materials, food and beverages [90,91]. This technique has superseded other speciation techniques like optical spectrometric, inductively coupled plasma atomic emission spectroscopy and atomic absorption spectroscopy which are less sensitive and do not provide simultaneous multi-elemental detection respectively as compared to ICP-MS. ICP-MS can detect trace-element-containing species, even when a particular trace element is distributed amongst a large number of species [92–94]. Ke-Lin et al. [66] also studied agricultural soils of Daxing, Beijing (north China plain) for metals Cu, Zn, Pb, Cr, Cd, Ni, As, Se, Hg and Co using this multiple element detection technique. Similarly, Zhang et al. [38] analyzed different heavy metals in soils under agricultural practices in Fuyang County, situated at the north of Zhejiang Province, near Hangzhou city, China. Abanuz et al. [95] monitored the contents of Cd, As, Pb, Zn, Mn, Cu, Cr and Hg in agricultural soils located in the vicinity of two large industrial zones i.e. the Gebze organized industrial zone (GOIZ) and Dilovası organized industrial zone (DOIZ) of Kocaeli city (Turkey). Identification and quantification of different heavy metals in soil mixture has been recognized by various researchers throughout the world using ICP-MS for its unique analytical capabilities [96–103].

2.4. Atomic fluorescence spectrometer (AFS)

Atomic-fluorescence spectroscopy (AFS) has undoubtedly become very sensitive, selective and versatile method for the determination of number of environmentally and biomedically important elements [81]. This technique has been used by several groups of workers as a complementary technique that may permit higher analytical sensitivity than atomic absorption spectroscopy

(AAS) for chemical analysis of various substances [104]. Comparative study of atomic fluorescence spectroscopy and inductively coupled plasma mass spectrometry for mercury and arsenic multispeciation was reported and it was suggested that although multispeciation based on ICP-MS exhibited better sensitivity than the coupling based on tandem atomic fluorescence but AFS was observed to be a very robust system with low cost of acquisition and maintenance. Due to its excellent sensitivity and selectivity, AFS was considered as one of the most important analytical tool for the analysis of trace elements, including mercury, arsenic and selenium by Gomez-Ariza et al. [105]. Yan-Feng et al. [106] analyzed heavy metals As, Hg, Cu, Zn, Pb, Cr, and Cd in soil of Wuxi city, Jiangsu Province, China under anthropogenic influence of heavy metal distribution using AFS. Speciation of different metals in soil mixtures have been explored by researchers throughout the world applying this technique [107–111].

2.5. X-ray fluorescence spectrometer (XRF)

X-ray fluorescence (XRF) spectroscopy is based on simple relation of the fundamental physics comprising of atom–radiation interaction. It is highly sensitive analytical tool used for the analysis of heavy metals [112–115]. Potts et al. [115] conducted quantitative analysis of rock samples by X-ray fluorescence spectrometry (XRF). They mentioned that major advantage of XRF was its ability to perform accurate quantitative analysis over a wide range of elements. However, they also mentioned that like most other analytical techniques, matrix effects (inter-element effects) were required to be corrected while performing quantitative analysis. Kaminski and Landsberger [116] reported Zn, Sb, Cd, Pb, Cu, Sn, As, Cr, Hg and Ni in soils (industrial and residential) with history of abundant industrial activities like smelters of ferrous and nonferrous metals, a coal-fired power plant, organic and inorganic chemical companies, municipal waste incinerators and petroleum refineries in East St. Louis, IL (Illinois, Urbana). Bhuyian et al. [117] reported Ti, Mn, Zn, Pb, As, Fe, Rb, Sr, Nb and Zr in soils of mine drainage and surrounding agricultural fields in Barapukuria coal basin located in Dinajpur District (northern part of Bangladesh) using X-ray fluorescence spectrometry. Cheng et al. [52] also estimated heavy metals Cu, Ni, and Zn in sediment soils from agricultural area in Taiwan contaminated due to waste pollution of industrial plants, such as electroplating, metalwork, textile, livestock, dyeing pharmaceutical, chemical plants, tanneries, and papermaking. Few studies have examined the utility of this technique for estimation of heavy metals in soils mixture [118–122].

2.6. Neutron activation analysis (NAA)

Neutron activation analysis (NAA) is a quantitative and qualitative method of high efficiency for the precise determination of trace elements in different types of samples. According to Soete [123] the mechanism of NAA was discovered in the year 1936 by Heves when he observed that the samples containing certain rare earth elements became radioactive on exposure to source of neutrons. The major difference between NAA and other techniques is that the former is based on nuclear transitions while latter ones on electronic transitions. Heavy metals were detected using epithermal neutron activation analysis by Alfassi [124]. He suggested that the important source for determination by activation analysis was the nuclear reactor due to its high flux of bombarding particles and radioactive capture reaction (n, γ) of thermal neutrons. During activation analysis, although the samples are activated mainly by n and γ, yet the whole spectrum of neutrons are used. He stated that the major drawback of this technique was the Compton's interference with the gamma ray of trace elements. Apart from this, some elements cannot be determined either due to too long half life of produced radionuclide or due to the reason that it does not emit gamma rays. However, epithermal neutron activation can overcome these difficulties. Determination of rare earth elements including La, Nd, Sm, Ti, Gd, Dy, Ho, Er, Lu including Th was done using thermal and epithermal neutron activation analysis was carried out by Brunfelt and Roelandts [125]. The major advantage of thermal and epithermal neutron activation analysis is that rare earth elements can be determined non-destructively by neutron activation using a short irradiation time. Neutron activation analysis is a sensitive multi-element analytical technique which is used for analysis of all types of elements including rare elements both qualitatively and quantitatively. Stafilov et al. [126] used NAA for estimation of Fe, Ti, As, Sb, Zn As, Au, Br, Co, Cr, Cu, Cd, Hg, Dy, Hf, In, Mn, Mo, Ni, Cd, Cu, Hg, In, Pb, Rb, Sb, Se, Sm, Sr, Tb, Th, U, V, W, Yb and Zn in soils from urban area, industrial zone, cultivable land, uncultivable land from polluted city of Veles region which is known for its lead and zinc industrial activity. Few scientific studies have reported the use of this analytical technique for determination of heavy metals in soil complexes [127–131].

2.7. D.C. argon plasma multielement atomic emission spectrometer (DCP-MAES)

It is validated analytical method for determination of several elements simultaneously. DCP-MAES system offers acceptable levels of selectivity, sensitivity, speed and economy for the analysis of many trace elements. Johnson et al. [132] determined the trace elements in natural waters using d.c. argon plasma multielement atomic emission spectrometer. Holmgren et al. [133] also estimated Cd, Pb, Zn, Cu, and Ni in agricultural soils at least 8 km downwind from any stack emitter (coal fired electric generator, smelter, foundry, etc.), 200 m from USA or state highways, 100 m from rural road ways, 100 m from current, abandoned, or known obliterated building sites, and 50 m from field boundaries.

2.8. Atomic absorption spectrometer (AAS)

Atomic absorption spectrometry (AAS) is the most common technique used for analyzing metals and some metalloids present in complex environmental matrices. AAS dates back to 19th century, when in 1950s Alan Walsh and his team developed this technique for determination of particular metal concentration within the test sample. This chemical technique can reliably analyze 62 different metals in a solution and has proven to be less time consuming, accurate and more convenient than other spectroscopic techniques for elemental analyses. The concentration is determined from calibration curve obtained by standards of known concentrations. The instrument uses flame and furnace spectroscopy for low detection limits and trace metal analysis [134–136]. Although recent scientific advances in other analytical techniques like ICP-OES, ICP-MS [137–147] have left AAS behind but still its larger specificity, makes it as a well established, excellent, reliable and most widely used chemical technique for analyzing any type of material [148–150]. It has been well reviewed and documented that this technique has been widely used by several workers from different regions of the world with an emphasis on soil heavy metal analysis [151–241].

2.8.1. Graphite furnace atomic absorption spectrophotometer (GFAAS)

Graphite furnace atomic absorption spectrophotometry (GFAAS) is a highly sensitive spectroscopic technique where samples are deposited in a small graphite tube which is heated to vaporize and atomize the analyte. The atoms after absorbing ultraviolet or visible light make transitions to the higher electronic energy shells and absorption is directly proportional to the concentration of the element. Thus with the help of standard graph curve, the concentration of the element can be evaluated. The major advantage of GFAAS over AAS is that the former provides excellent detection limits for measuring concentrations of metals in aqueous and solid samples. Apart from this, GFAAS is helpful to detect very few concentrations (even upto ppb levels) of heavy metals. Sun et al. [71] estimated cadmium in needles and rooting soils of Masson pine trees growing along an urban–rural gradient in Guangzhou, China, using graphite furnace atomic absorption spectrophotometer. Many other authors over the world have adopted this technique to estimate different heavy metals in various soil samples [242-252].

2.9. *Scanning electron microscope with energy dispersive X-ray (SEM–EDX).*

Although SEM-EDX is another technique to detect heavy metals yet it is rarely permitted or used to detect heavy metals or rare earth metals in soil matrix. However, heavy metal rich minerals can be identified by SEM-EDX provided that all soil sub-portions uniformly contain the heavy metal content [253]. Mineralogical analysis using SEM with EDX was conducted by Palumbo et al. [245]. The analysis was performed on high density sand fraction of samples using Na–tungstate saturated solution (SG $>$ 2.96 g/cm) as well as different pedons. The heavy sand fraction, analyzed by SEM/EDX was found to be enriched with iron oxides while TR pedon was found to contain abundant manganese nodules. The authors further conducted chemical analysis by using AAS with graphite furnace to estimate other heavy metals. The advantage of SEM–EDX is that the sample can directly be analyzed without carrying the digestion procedure provided the element/s is/are uniformly distributed in soil sample. As only grain or small fraction of soil can be analyzed with SEM–EDX, it cannot detect the element, if it is inconsistently distributed in a pedon.

3. Conclusion

Soil ecosystem receives different contaminants including heavy metals through various natural as well as anthropogenic sources such as application of agrochemicals, release of sewage effluents and vehicular emissions. These heavy metals pose serious health risks upon entering human body *via* inhalation, ingestion or skin contact. Therefore, the concentration of heavy metals must be monitored from at regular intervals in soil matrices in order to keep a check on pollution levels. There are many established analytical techniques like inductively coupled plasma optical emission spectroscopy (ICP-OES), inductively coupled atomic emission spectroscopy (ICP-AES), inductively coupled mass spectroscopy (ICP-MS), atomic fluorescence spectrometry (AFS), X-ray fluorescence spectrometry (XRF), neutron activation analysis (NAA), d.c. argon plasma multielement atomic emission spectrometry (DCP-MAES), scanning electron microscope with energy dispersive X-ray (SEM–EDX) and atomic absorption spectrometry (AAS) for the purpose of quantification of heavy metals in soil. However, regardless of advantages of other techniques during this study, Atomic Absorption Spectrometry was found to be the most widely used technique due to its sensitivity and wide detection limits varying from parts per trillium (ppt) to parts per million (ppm) levels. The present review focuses on various digestion methods, use of analytical techniques and concentration of different heavy metals in soil samples collected from different regions of the world.

Appendix A. Supplementary materials

Supplementary materials associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.talanta.2014.02.033.

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