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ORIGINAL PAPER

Assessing cadmium risk in wheat grain using soil threshold values

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Abstract At present, the prior-established threshold values are widely used to classify contaminated agricultural soils with heavy metals under the cultivation of a variety of crops, without considering the different sensitivity of plants to heavy metals. Evaluation of the characteristics of cadmium transfer from a polluted calcareous soil to cultivated wheat crop and assessment of the efficiency of using the threshold values to reflect the soil pollution risk by cadmium in Zanjan Zinc Town area at the northwest of Iran were the goals of this study. Totally, 65 topsoil (0–20 cm) and corresponding wheat samples of an agricultural region in the proximity of a metallurgical factory were collected and analyzed for cadmium concentration. The results revealed that industrial activities strongly control cadmium distribution in the studied soils. Relatively high bioavailable cadmium contents (mean 0.77 mg kg^{-1}) were found in the soils, notwithstanding their alkalinity. It was observed that just 22.5% of the studied area around the Zinc Town is covered by polluted soils with the cadmium concentration exceeding the maximum permissible concentration of 5 mg kg^{-1}, whereas cadmium concentration in wheat grains of 19 sampled plants is higher than the threshold value of 0.2 mg kg^{-1}. Among these polluted plants, a total of eight samples were grown in areas classified as unpolluted soils with cadmium, based on the soil threshold value. It seems that this misclassification of polluted soils is mainly related to the crop sensitivity to heavy metals uptake from the soil which should be considered.

Keywords Food safety · Heavy metals · Risk assessment · Soil quality standards · Zanjan Zinc Town

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Introduction

Contamination of agricultural soils with heavy metals (HMs) has attracted increasing attention worldwide (Rizwan et al. 2016). HMs as one group of the most dangerous contaminants for living organisms may enter the soil by natural processes or a wide range of human activities (Wang et al. 2013). A good understanding of HM sources and their spatial distribution is the prerequisite for controlling their numerous hazardous effects on ecosystem (Feng et al. 2012). Soil pollution by HMs is commonly assessed by mapping the interpolated values of HMs concentrations through a given area. Regulations to protect humans from toxicities related to HMs are primarily based on soil quality references or threshold values (Manga et al. 2014). Accordingly, productive soils are divided into uncontaminated (appropriate for crop farming) and contaminated (not appropriate for crop farming) classes by farm managers (Liu et al. 2015).

This classification system may have some unavoidable problems which may lead to the site misclassification. First of all, the threshold values are generally given based on the total concentration of HMs in soils for specific conditions, due to the fact that different soil properties seriously affect HM behavior in the soil (Manga et al. 2014). Soil factors, including pH, clay type and amount, organic matter (OM), and cation exchange capacity (CEC), are considered as the most important properties which influence the mobility and availability of HMs for living organisms in the soil (Safari-Sinegani and Jafari-Monsef 2016). So far, inconsistent results have been reported in the studies performed on the roles of various soil properties in the determination of the toxicity and bioavailability of HMs (Luo et al. 2014). Furthermore, these properties may have short-range variations in the field, causing difficulties for the assessment of their effects on HM behavior (Vega et al. 2010). Consequently, regarding a unique threshold value for a specific HM in different locations in the whole field seems to be not acceptable.

The other serious problem about the above-mentioned classification system arises from the fact that HM risk assessment for humans, especially in agricultural soils, is influenced by another important factor, plant species (Kabata-pendias 2010). Particularly, human's life is threatened by contaminated food crop consumption because of contaminated environments caused by HMs (Liu et al. 2015). Plant ability to uptake HMs from the soil is mainly influenced by plant species, and it varies between different varieties of the same plant (Kubo et al. 2016). Therefore, under the same soil conditions, any metal and plant interactions occur specifically (Liu et al. 2009). Accordingly, it is recommended to evaluate the health risk of soil pollution by HMs by investigating the characteristics of heavy metal transfer from soils to plants for each particular soil–plant system (Lopes et al. 2012).

As a major food plant throughout the world, wheat (*Triticum aestivum* L.) is able to take up varied HMs by its roots (Kubo et al. 2016) and transfer them to the areal parts, especially grains (Wang et al. 2013). International organizations, like FAO and WHO, set threshold values of different HMs in various food materials, especially wheat grains (FAO/WHO 2012). Accordingly, probability of exceeding HM contents in wheat grains than their threshold values should be considered in designing the land use management strategies in contaminated soils (Liu et al. 2015).

Cereal crops, particularly winter wheat, are extensively grown in the fertile agricultural lands around the Zinc Town in the south of Zanjan City, northwestern Iran. Due to the probable addition of different HMs, especially cadmium (Cd) to the adjacent soils via industrial activities in the Zinc Town (Saba et al. 2015), Cd transfer from surface soil to cultivated wheat plant and pollution of wheat crop by this hazardous element are very likely, unlike the soil alkalinity and high contents of calcium carbonates in the soil. Cd is considered as one of the most dangerous HMs that threatens agricultural productivity, food safety, and consequently human health (Rizwan et al. 2016). However, to assess soil pollution risk by HMs in such a contaminated agricultural land, setting the threshold values is the most common procedure all over the world, unlike its deficiencies (Moreno et al. 2009; Manga et al. 2014). Therefore, studying Cd transfer from a polluted calcareous soil to wheat crop and evaluation of the efficiency of using threshold values to reflect the real risk of soil pollution by Cd were the main purposes of this research.

Materials and methods

Study area

The study area of Zanjan plain is 100 km^2, which is located to the southwest of Zanjan City at northwestern Iran (Fig. 1). This area is located close to a large metallurgical factory, leading to soil pollution by different HMs, especially Cd. Cereal crops, especially winter wheat, are being extensively cultivated in the cropland. The immature studied soils are relatively shallow and the calcium-carbonate-rich parent material in this area has caused an alkaline soil pH. The mean annual rainfall and temperature in Zanjan region are 302.8 mm and 11.1 °C, respectively, and the average elevation of studied area is about 1660 m above sea level.

Sample collection and physicochemical analyses

In total, 65 sampling sites were selected in all of the lands that were under wheat cultivation at the time of study as shown in Fig. 1. At each sampling site, the surface soil (0–20 cm), in which wheat roots were mainly distributed (Nan et al. 2002), and corresponding wheat samples were taken in July 2014, just before the wheat harvest. The soils were air-dried and mildly ground to pass through a sieve with 2 mm round holes before analyzing their physicochemical properties by using standard methods (Soil Survey Staff 2014). The soil samples were submitted for total Cd concentration analyses using HCl–HNO_3 (Sposito et al. 1982). By employing the method of diethylenetriamine-penta-acetic acid (DTPA), the available Cd was extracted from the soils and determined using an atomic absorption spectrometer (Lindsay and Norvell 1978). Plant tissues (grains, roots, and leaves and shoots) were well washed with running tap water and distilled water to remove soil particles from the plant surfaces. All the tissue samples were dried at 65 °C for 48 h, well ground, and homogenized to pass through a 0.4-mm polyethylene sieve. The

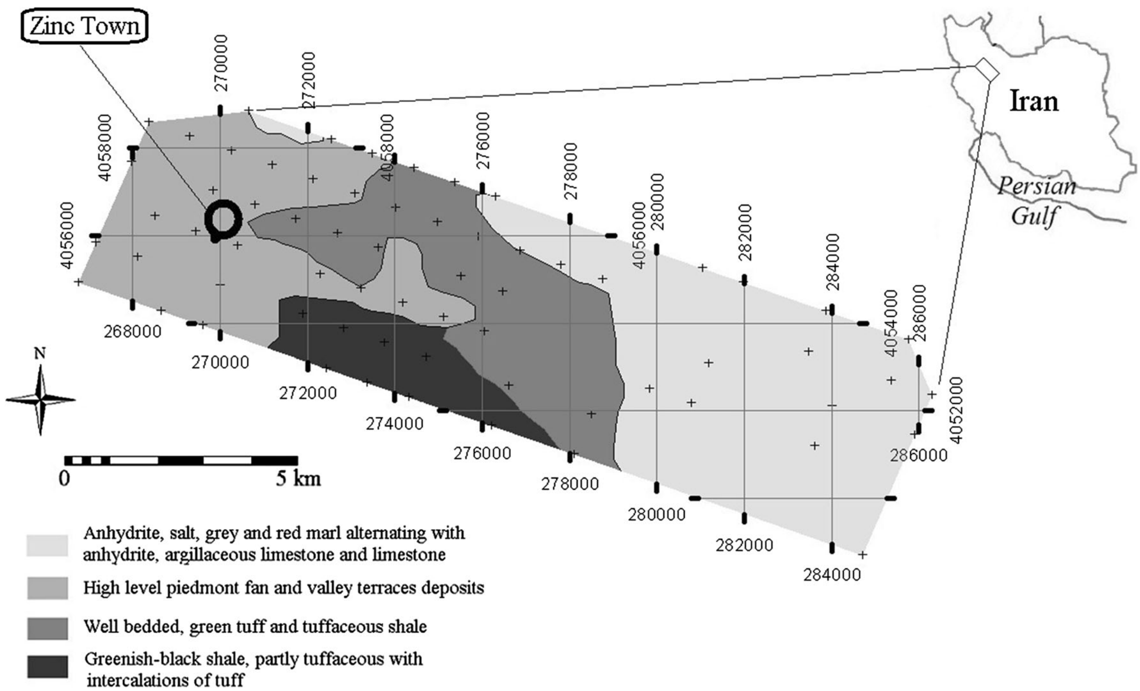

Fig. 1 Distribution of 65 sampling points in Zanjan plain with the geologic map of the study area, northwest Iran

powdered plant samples were subjected to digestion in a solution of 70% aqua regia (HNO_3 + concentrated HCl) and 30% H_2O_2 and then analyzed for Cd by graphite furnace atomic absorption spectrometry (Westerman 1990).

Statistical and geostatistical analyses

Statistical analysis of the data was done by using SPSS software (ver., 19.0, SPSS Inc., USA). To determine how Cd bioavailability in studied soils and its transfer to wheat grain are influenced by soil properties, correlation analysis was applied. The bio-concentration factor (BCF), as Cd concentration ratio of wheat roots to the bioavailable Cd concentration in the soils, and the translocation factor (TF), as the Cd concentration ratio of wheat grain to the wheat root, were calculated to study the relative ability of cultivated wheat plant to take up Cd from the soil and transfer it to grains. The ordinary kriging estimator was used to interpolate soil total and bioavailable Cd concentration at unsampled locations and finally, the spatial distribution maps of Cd contents in the soil were prepared using ArcGIS software (ver. 10.2; ESRI). The mapping units of soil total Cd concentration map were separated based on two threshold values, proposed by Department of Environment of Iran (2013): the maximum permissible concentration (MPC), soils having Cd concentration above which are not considered as suitable lands for agricultural uses, and the clean-up concentration, soils having Cd concentration above which should be refined immediately to prohibit the pollution spreading. It should be noted that most soils in Iran are of a calcareous type having moderately alkaline conditions (pH between 7.4 and 8.8). Regarding relatively strong effects of soil pH and calcium carbonates on HM behavior in the soil (Ming et al. 2016), the proposed MPC and clean-up values by Department of Environment of Iran (2013) for Cd are higher than the common threshold values in other countries (5 and 20 mg kg^{-1}, respectively). To assess the efficiency of using these threshold values for precise classification of soils in relation to Cd transfer to food chain, exceeding the threshold Cd content of 0.2 mg kg^{-1} in the wheat grain (FAO/WHO 2012) was assessed, considering the total Cd concentration in corresponding soil.

Results and discussion

Relevant soil properties

The soil properties analyzed in the study area are shown in Table 1. Through the K–S test, it was verified that the selected soil properties were normally distributed. Soil OM content was very low (mean < 1%), which may reflect the immaturity and youthfulness of the studied soils. Relatively high contents of calcium carbonate equivalent (CCE) were observed in the studied soils (ranging from 5.8 to 33.0%), which is the dominant property of the Iranian soils, especially in semiarid regions of northwest Iran (Qishlaqi et al.

Table 1 Summarized statistics of the soil properties selected

Variable	Mean	Minimum	Maximum	Median	SD	Coefficient of variation (%)	K–S p^a
OM (%)	0.69	0.45	1.12	0.66	0.18	25.54	0.38
Clay (%)	26.20	10.00	37.00	28.20	6.29	26.48	0.50
CCE (%)	16.94	5.80	33.00	15.25	6.99	41.30	0.32
pH	7.73	7.45	7.93	7.74	0.66	8.57	0.58

[a] K–S p: the significance level of Kolmogorov–Smirnov normality test

2009). These high contents of CCE seem to enhance soil pH, causing relatively alkaline conditions in the studied soils (soil pH range of 7.45–7.93).

Soil Cd concentration

Statistical analyses

The total soil concentrations of Cd ranged between 0.80 and 42.30 mg kg^{-1} with an average of 5.14 mg kg^{-1} (Table 2). It is reported that Cd commonly co-occur with Zn compounds in the natural environment; in turn, metal refining of Zn has resulted in large amounts of wastes containing both HMs (Ming et al. 2016). The median index for soil total Cd concentration, however, is much smaller than the average, implying that Cd distribution in the studied soil is influenced by few high values (contamination hotspots).

Bioavailable (DTPA-extractable) Cd concentration in the studied soils ranged between <DL and 5.2 mg kg^{-1} (average = 0.77 mg kg^{-1}) (Table 2). It is widely accepted that most of the metals in soil have a fairly strong binding with organic or inorganic compounds and so, just small fractions of them may enter into the soil solution as the readily available fraction for plant uptake (Kabata-pendias 2010). Therefore, total concentration of HMs in soil is just one of the numerous factors which may influence their bioavailable concentration (Wang et al. 2013), whereas their natural or anthropogenic sources (Kabata-Pendias 2010), their interactions in soil environment (Liu et al. 2009), land use type, the duration of their contacts with the soil compounds and to a greater extent, relevant soil properties may be more determining in this respect (Vega et al. 2010). Specifically, Cd mobility and availability in soil has a large impressibility from soil OM (Moreno et al. 2009). Accordingly, the OM in studied soils can be partly responsible for fairly high Cd bioavailable concentration observed in the present study.

Cd spatial distribution in the soil

Figure 2 shows the spatial distribution of total and bioavailable soil Cd concentration. As shown in Fig. 2, the point source pollution, i.e., Zinc Town, mainly controls the Cd distribution in the studied soils, so that both of the total and bioavailable soil Cd contents reduced with a greater distance from the Zinc Town. Consequently, 22.5% of the studied area around the Zinc Town is covered by polluted soils with total soil Cd concentration exceeding the MPC of 5 mg kg^{-1}. The extensive similarity between the spatial distribution of total and bioavailable soil Cd concentration may confirm that Cd bioavailability in the studied soils is more influenced by its total concentration, in turn, by the Zinc Town, compared with soil properties. Some slight differences were found, which may arise from the controlling effects of soil properties on Cd bioavailability. Saba et al. (2015) reported that the industrial activities in Zinc Town may cause the accumulation of different HMs in the surrounding soils, via the precipitation of suspended materials in emissions or reflowing of dumped tailings by wind, rain, and runoff actions.

Cd transfer to the wheat plant from the soil

Table 3 shows the summarized statistics for Cd concentrations in the different parts of wheat plants and those of TF and BCF indices. Cd concentration range within the cultivated wheat roots was between <DL (0.01 mg kg^{-1}) and 4.60 mg kg^{-1} with an average of 0.46 mg kg^{-1} (Table 3). It is reported that among different HMs, Cd is

Table 2 Cd concentration in the studied soils (mg kg^{-1})

Variable	Mean	Minimum	Maximum	Median	SD	Coefficient of variation (%)	K–S p^a
Near-total Cd (mg kg^{-1})	5.14	0.80	42.30	2.70	7.47	145.28	0.00*
DTPA-extractable Cd (mg kg^{-1})	0.77	<DL[b]	5.20	0.25	1.16	151.29	0.00*

* $p < 0.05$

[a] K–S p: the significance levels of Kolmogorov–Smirnov normality test

[b] DL: detection limit for atomic absorption spectrometry

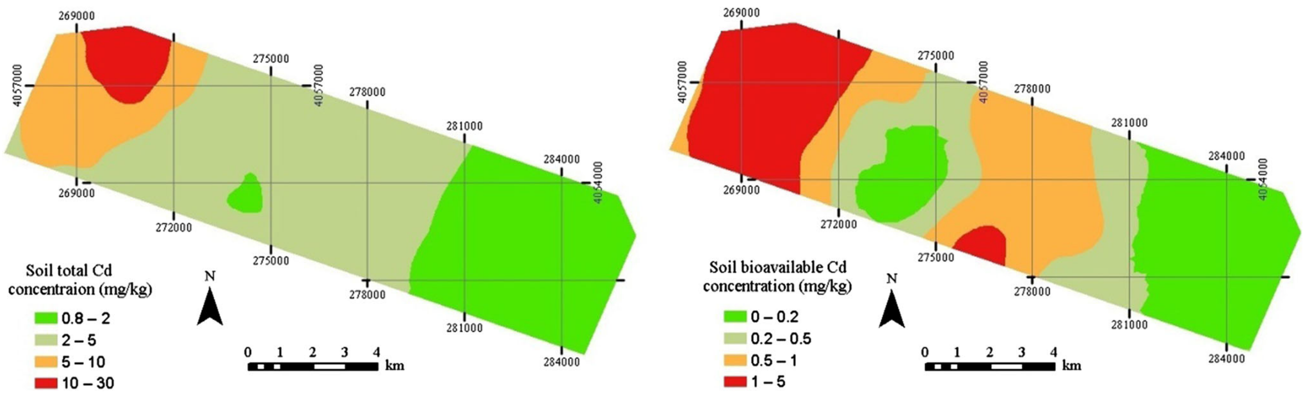

Fig. 2 Spatial distribution of total (*left*) and bioavailable (*right*) soil Cd concentration

Table 3 Cd content in each part of wheat plant (mg kg^{-1})

Variable	Mean	Minimum	Maximum	Median	SD	Coefficient of variation (%)	K–S p[a]
Root Cd	0.46	<DL	4.60	<DL	0.94	203.8	0.00*
Shoot Cd	0.35	<DL	4.10	<DL	0.76	214.54	0.00*
Grain Cd	0.27	<DL	2.85	<DL	0.60	213.10	0.00*
BCF	0.60	0.00	1.54	0.65	0.50	82.68	0.08
TF	0.55	0.00	1.25	0.50	0.34	62.08	0.87

* $p < 0.05$

[a] K–S p: the significance levels of Kolmogorov–Smirnov normality test

one of the more mobile elements, especially at the absence of OM (Safari-Sinegani and Jafari-Monsef 2016). It seems that low OM in the studied soils (Table 1) provides a good medium for Cd transfer through the soil and finally reaching wheat plant roots. Out of that, owing to its chemical similarity to microelement Zn, Cd is often taken up in biochemical pathways similar to Zn (Ming et al. 2016). The relatively vast range of calculated BCF values (Table 3), however, may indicate the selective uptake of Cd by wheat roots through the field, based on other influencing factors.

Cd concentration in the wheat shoots had a range between <DL and 4.10 mg kg^{-1} with an average of 0.35 mg kg^{-1} (Table 3). Since the harvested wheat shoots in Zanjan region will be directly used as animal food, these relatively high Cd concentrations in wheat shoots could result in some serious problems in target organs, posing a significant health risk to humans. It is widely believed that most of the nonessential elements adsorbed by plants remain in plant roots (Kabata-pendias 2010). Specifically, wheat roots commonly serve as a barrier to HM translocation to the above parts of the ground, through Cd retention by root cell vacuoles (Liu et al. 2009). It is suggested that the high retention of Cd in wheat roots may be caused by chelation with organic acids (Adeniji et al. 2010). By contrast, some researchers believed that wheat roots easily take up the Cd present in the soil and translocate it to the edible parts (Kubo et al. 2016; Rizwan et al. 2016). It seems that fairly high BCF and TF values in the present study (Table 3) may support the latter idea.

The effects of soil properties on Cd transfer to wheat grains

Table 4 represents Pearson's correlation coefficients between Cd concentrations and soil parameters.

Soil pH, OM content, and type and content of clay minerals in soil are listed as the major soil properties with respect to Cd availability in the soils and therefore Cd uptake by plants roots (Vega et al. 2010; Ming et al. 2016).

Table 4 Correlation coefficients between Cd concentrations and soil properties

Variable	OM	pH	Clay	CCE	Distance from Zinc Town	Logged near-total Cd	Logged DTPA-extractable Cd
Logged DTPA-extractable Cd	−0.012	−0.110	−0.162	−0.383**	−0.386**	0.789**	1
Logged grain Cd	−0.017	−0.173	−0.056	−0.357**	−0.368**	0.910**	0.736**

** Correlation coefficient is significant at the level of 0.01 (2-tailed)

Correlation analyses revealed that these factors are poorly correlated with bioavailable Cd concentration in studied soils and its transfer to wheat plant (Table 4). These may result from the limited range of pH values (Qishlaqi et al. 2009), the low contents of soil OM (Jalali and Moharrami 2007), and probably the absence of highly weathered clay minerals with high Cd absorption capacity, like Fe and Mn oxides in studied soils (Vega et al. 2010), respectively. On the other hand, Cd availability in the studied soils and its transfer to wheat plants were negatively correlated with CCE contents in studied soils (Table 4). It is believed that carbonates may effectively immobilize HMs due to surface adsorption or precipitation mechanisms (Jalali and Moharrami 2007). Safari-Sinegani and Jafari-Monsef (2016) have confirmed that HMs availability in calcareous soils are strongly controlled by CCE contents in soils.

Out of the soil properties, correlation analyses showed that bioavailable Cd concentration in studied soils, as well as Cd transfer to the wheat grains from the soil, is positively influenced by the soil total Cd concentration (Table 4). This may infer that the increment of soil total Cd content could increase Cd accumulation in cultivated wheat grain under field conditions. It is reported that Cd uptake by different plants may be enhanced at the elevated concentrations of other HMs, especially Zn and Pb, in soils (Nan et al. 2002). Regarding the fairly high Pb concentration in studied soils (Saba et al. 2015), this chemically similar cation to Cd may simplify the Cd movement through the soil by occupying the exchange sites and finally lowering the soil absorption capacity for Cd cations (Nan et al. 2002). Furthermore, it seems that soil components have had insufficient time to immobilize a significant amount of newly added industry-derived Cd to the studied soils (Parizanganeh et al. 2010). Consequently, disregarding the bioavailable Cd concentration in the studied soils, total concentration of the soil could be reliably used for estimating the Cd transfer rate from the underlain soil to the cultivated wheat grain. These findings were in line with the previous reports (Ding et al. 2013; Wang et al. 2013).

The distance from Zinc Town was recognized as another important factor inversely influencing bioavailable Cd concentration in studied soils and Cd contents in cultivated wheat crop (Table 4). It is reported that fine aerosols, containing various metals, may travel long distances floating in the air (Dimitrijevic et al. 2016). However, based on climatic conditions, most of these particles finally fall out on the underlying soil by wet or dry precipitations, near or not too far from their industrial sources (Kabata-pendias 2010). Parizanganeh et al. (2010) reported that the extremely high levels of metals in the dumped tailings near the Zinc Town indicated a potentially dangerous source of pollution in the adjacent lands. Furthermore, it seems that high mountains around the Zinc Town may act as a natural barrier for areal particles movement, leading to stimulate the aerosols precipitation in soils near the industrial pollution source, i.e., the Zinc Town. These results were in accordance with the previous investigations, in which the concentrations of HMs in soils and cultivated crops reduced with distance from the main pollution source (Feng et al. 2012; Dimitrijevic et al. 2016).

Cd risk assessment with regard to the soil threshold values

The risk of Cd transferring was assessed based on the probability of its relocation from the soil to wheat grain in values higher than the maximum allowable content of 0.2 mg kg^{-1}. Assessing the efficiency of using the soil threshold value (the MPC of 5 mg kg^{-1}) to reveal the real risk of Cd in studied soils with respect to wheat pollution with Cd, its content in wheat grains was analyzed considering the total soil Cd concentration in corresponding soils (Fig. 3). As shown in Fig. 3, Cd concentrations in the wheat grains of 19 sampled plants were higher than the threshold value of 0.2 mg kg^{-1}. These findings clearly showed that appreciable amounts of harvested crop (29.2%) contain hazardous amounts of Cd, which may cause health problems for local consumers. Regarding the relatively low Cd concentration in the root part of sampled plants (Table 3), it seems that cultivated wheat can simply transfer significant amounts of taken up Cd from roots to grains. It is suggested that Cd is directly transported from wheat root to its grain via the stem through the grain filling period (Rizwan et al. 2016).

Polluted wheat samples with Cd were mainly grown in areas classified as polluted soils with Cd, based on the MPC of 5 mg kg^{-1}. However, among these 19 polluted wheat samples, 8 samples (highlighted with red points) were grown in the soils containing total Cd concentration less than MPC (Fig. 3). Regarding the obvious fact that the MPC for HMs in soil is mainly based on the lack of

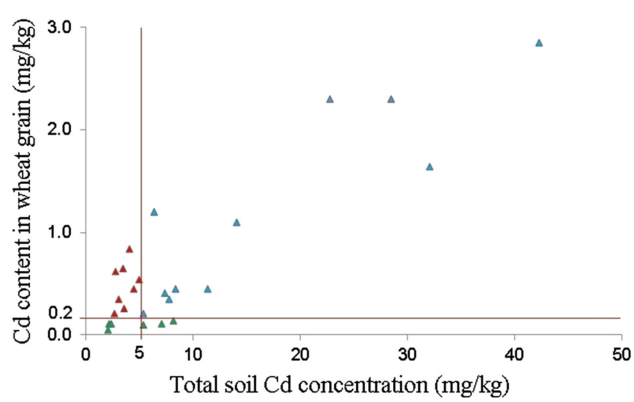

Fig. 3 Relation between soil total Cd concentration and Cd content in wheat grains

permission to any contaminations of the food chain (Liu et al. 2015), this observation may indicate that the suggested MPC value for calcareous soils of Iran could not accurately reflect the Cd risk. Lubben and Sauerbeck (1991) observed that wheat grain with Cd contents lower than the threshold value of 0.12 mg kg^{-1} was found just with Cd concentrations of the soil lower than 0.5 mg kg^{-1}, whereas the MPC of Cd regarded in the soil was 5 mg kg^{-1}. Similar results were reported by Salazar et al. (2012), who reported, even for lower soil Cd concentration than the maximum permissible level, more than the threshold value of 0.2 mg kg^{-1} Cd was found in soybean grown on field conditions.

The observed misclassification of polluted soils in the present study is an unavoidable result of not considering the land use types at the time of establishing the threshold values. In other words, it seems that relatively high capability of studied wheat cultivar for Cd uptake, reflected by fairly high BCF contents (Table 3), may explain the observed site misclassification. Therefore, if the suggested MPC was being assessed for another less sensitive crop to Cd uptake, it probably had a better performance. Rizwan et al. (2016) found that the threshold of phytotoxic concentrations of Cd varies even with wheat genotypes. However, it should be noted that the accumulation prediction model for the plants fitted by the total Cd of the soil often had a better performance compared to the models computed by the exchangeable Cd (Nan et al. 2002; Ding et al. 2013).

Although other researchers have reported the inefficiency of separating polluted soils from unpolluted ones based on the threshold values (Manga et al. 2014; Liu et al. 2015), they extensively focused just on the defects arising from the inaccurately assigned importance to the numerous soil parameters controlling the bioavailability of HMs in the soil (Moreno et al. 2009). Considering relatively low correlation between the bioavailable Cd concentrations and the studied soil properties (Table 4) and also the magnitude of suggested MPC, it can be stated that this defect may not be responsible for the observed misclassification in the present study. Our findings, however, indicate that plant species should be considered as an important factor by land managers for establishing the precise threshold values.

Conclusion

Analyses of Cd concentration in the studied soils and cultivated wheat plant indicated that Cd contents and its spatial distribution in both environmental components is highly affected by metal processing activities in Zinc Town. Among the different wheat plant parts, the roots had the highest Cd concentration, with an average of 0.46 mg kg^{-1}, whereas Cd concentration in wheat shoots and grains gradually decreased, with an average of 0.35 and 0.27 mg kg^{-1}. Correlation analyses revealed that Cd bioavailability in the studied soils and its transfer to wheat grain are negatively related to the distance from pollution source and calcium carbonate contents. Therefore, it is suggested that assigning the adjacent agricultural lands to another type of land use may be the most secure way in order to avoid the deleterious effects of Cd pollution caused by industrial activities in Zinc Town on public health. Among the 65 harvested wheat samples, a total of 19 polluted samples (containing a higher Cd concentration than the threshold value of 0.2 mg kg^{-1}) were found, while 8 of them were grown in areas covering unpolluted soils, based on the MPC of 5 mg kg^{-1}. It can be concluded that establishing the threshold values to assess the soil pollution risk by HMs just based on the soil properties without considering the target crop may result in unsatisfactory results. Accordingly, it is suggested that a specific threshold value should be considered for each especial plant based on its ability for taking up the HMs from the soil.

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