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ORIGINAL PAPER

Evaluation of spinach (*Spinacia oleracea*) quality depending on various combinations of soil and water attributes

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Abstract
The aim of the current study was to investigate the effects of quality of soil and water on spinach (*Spinacia Oleracea*) quality, cultivated in district Dera Ismail Khan, Pakistan, with respect to some heavy metals. For this purpose twelve samples each of water, soil and spinach were collected from four different localities of the district. Out of two sites were irrigated with tube well water (DIK Cantonment and Hathala Kulachi area), third one with canal water (Paroa area) and the fourth one with sewage water (Dappawala band area, Indus River). The quality of water and soil samples was analyzed for pH, EC, TDS, and mineral contents to assess their impact on the minerals uptake by spinach. Metals, i.e., sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), zinc (Zn), iron (Fe), copper (Cu), lead (Pb), and nickel (Ni) were analyzed via atomic absorption spectrophotometer. The maximum values of metals were recorded for sewage water site followed by canal water and least for tube well irrigated sites.

Keywords Spinach · Sewage water · Mineral uptake · Atomic absorption spectrophotometer · Pollution index

Introduction

In some ways, water contamination is more challenging than environmental pollution. Anything can be poured or discarded into water, from domestic and industrial effluents (Yan et al. 2021). As a result, it lacks the quality necessary to irrigate any type of crop and contains numerous poisonous chemicals. Numerous heavy metals are present in vegetables grown with sewerage water, posing serious health hazards to both humans and animals (Yu et al. 2018).

The control of soil productivity is a different issue that farmers deal with. The ability of soil to produce crops in practical quantities is known as productivity, and it is influenced by the soil's physical and chemical features as well as the meteorological and hydrological systems in which it is located (Yin et al. 2019). Globally, the decline in soil productivity has been a persistent worry. Sewage water can increase the production of leafy vegetables in the short term; however, continued use has a negative impact on the soil's economic output (Hou et al. 2021); (Hussain et al. 2011; Punz and Seighardt 1993; Bahemuka and Mubofu 1999; Alam et al. 2003; Sharma et al. 2006; Perveen et al. 2012).

Among all the food crops green leafy vegetables occupy central position as they provide ample amount of vitamins and minerals. *Spinacia oleracea Linn* is commonly known as "Spinach" which belongs to Amaranthaceae family. It contains huge amount of core nutrients; both macro and micronutrients (like Mg, Na, K, P, Ca, Cu, I, Zn, Mn, and Fe etc.) and several vitamins (A, E, K, C, and B complex). It is also rich in folic acid, carbohydrates, proteins, amino acids, fiber, and lipids thus referred as "powerful and popular food" (Perez Espinosa et al. 1999; Tandi et al. 2004; Segheloo et al. 2014; Tehseen et al. 2014; National Osteoporosis Foundation, 2015). Therefore, spinach is not only used for improving nutritional status in human but also used as an anti-anemic, anticancer, anti-inflammatory, analgesic, and

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antifungal agent. It is also used in lowering of blood pressure and for treating bowels and dysentery (Roshan et al. 2012).

Food chain contamination with heavy metals is a burning issue all over the world (Lokeshwari and Chandrappa 2006). From the environment, harmful heavy metals assemble in the crops through different routes such as water and soil pollution. Deposition of heavy metals at elevated level can deteriorate the quality of crops (Mielke and Reagan 1998; Mielke et al. 1999; Andrews et al. 2002). The heavy metals after bio-accumulation in the food crops, transfer to human and animal body via food chain and cause several diseases (Lokeshwari and Chandrappa 2006; Mahmood et al. 2020).

A number of studies have been conducted on food chain contamination with heavy metals (Balkhair and Ashraf 2015, Lawal and Audo 2011; Asgharipour and Azizmoghaddam 2012; Kamal et al. 2013; Kumar et al. 2016; Zakir et al. 2018), including in Pakistan (Lone et al. 2003; Khan et al. 2010; Naz et al. 2018; Riaz et al. 2018; Perveen et al. 2012; Mahmood et al. 2020) but very few correlate the composition of water, soil and the vegetable/ fruit. The current research was aimed to investigate the impact of quality of water used for irrigation and soil on the concentration of heavy metals uptake by spinach cultivated in Dera Ismail Khan, Pakistan.

Materials and methods

Methodology

Current study was performed in district Dera Ismail Khan, Khyber Pakhtunkhwa Province, Pakistan. Dera Ismail Khan is located at 31° 49′ 53.3352" N and 70° 54′ 41.7528" E with an area of 9,334 km^2 having total population of 1,627,132 (according 2017 census). It is situated at 178 m (583 ft) above the sea level. The samples of water, soil and spinach were collected from four different sites (Fig. 1), namely (1) Army Dairy farm situated at Dera Ismail Khan Cantonment, irrigated with Tube well water, (2) Area irrigated by Tube well # 49 water, located near Hathala, Maddi, Kulachi, (3)

Fig. 1 Map of the sampling sites

From Paroa area irrigated with Chashma Right Bank canal (CRBC) and (4) The area near slaughter house Dappawala band, Indus River irrigated with city effluent/ sewage water of Dera Ismail Khan. Totally 48 samples were collected for calculation of physical parameters and evaluation of heavy metal concentration. The GPS location of the site is Hathala (32.06038, 70.59491), Dera Ismail Khan Cantt. (31.82610, 70.92491), Dera Ismail Khan dup band area (31.79251, 70.90896), Paroa (31.56547, 70.72129). The history about the application of fertilizers and water used for irrigation was also collected from the farmers.

Sampling

All the water samples were collected in new screw capped labeled plastic bottles pre-washed thrice with the same water sample. Soil samples were also collected from the field (as mentioned in the methodology portion) from the depth of 0–30 cm, and kept in labeled brown paper bags sealed appropriately. Similarly, the leaves of spinach samples were randomly collected from the same field, kept in labeled brown paper bags sealed appropriately. All the samples were transported to the laboratory immediately where these were stored for analysis. The water samples were stored at room temperature for further analysis. The soil samples were kept in labeled stainless steel trays for drying at room temperature for few days. However, one gram of each soil sample was kept in weighed watch glass and put in oven at 120 °C for complete drying for the measurement of moisture contents. The remaining soil after drying, was ground, sieved through 2 mm sieve and finally stored at room temperature in a screw capped labeled plastic jars for further analysis. The spinach samples were thoroughly washed with tap water and then with distilled water to remove the dust/dirt or soil particles and left overnight at room temperature for drying. On the next day, the weight of each washed sample was measured and then dried in an oven at 60 °C till its dryness. The samples were cooled down to ambient temperature and then grinded in an electric grinder into fine powder, sieved through 2 mm sieve and finally stored in a screw capped labeled plastic jars for further analysis.

Statistical analysis

The statistical analysis of data was performed by using Statistical Package for Social Sciences software version 22 (SPSS). The relationship between metals were find out by applying *t*-Test.

Sample analysis for physicochemical parameters

Water analysis

After sampling the water samples were immediately analyzed for pH, turbidity, electrical conductivity, and total dissolved solids (TDS) by following the methods described by Chaurasia and Gupta (2014). The detailed analysis is provided as supplementary information (ESI).

Soil analysis

The soil samples were analyzed for pH, electrical conductivity, and moisture contents by following the methods described by (Chaurasia and Gupta 2014). The detailed analysis is provided as supplementary information (ESI).

Plant analysis

Spinach samples were analyzed for moisture contents by following the methods described by Chaurasia and Gupta (2014).

Solution preparation of water and soil samples

Water and soil samples were analyzed for heavy metals and micro/macro nutrients like Na, K, Ca, Mg, Zn, Fe, Cu, Pb, Ni by following the methods described by Isaac and Kerber

Table 1 Physical parameters of water used for irrigation

Parameters	Tube well water (DIK Cantonment) ($n = 12$)	Tube well water (Hathala, Kulachi) ($n = 12$)	CRB Canal water (Paroa) ($n = 12$)	City sewage water (Dappawala band area) ($n = 12$)	Standard upper limit for irrigation*
Temperature(°C)	18.3	17.7	12.5	20	–
pH	7.5(±0.543)	7.2(±0.764)	7.02 (±0.663)	8.67(±1.43)	6.5–8.4
Turbidity(NTU)	0.99(±0.102)	1.5(±0.236)	2.5(±0.236)	2.76(±0.576)	2–5
EC (dS/m)	1.651(±0.1203)	1.023(±0.057.23)	2.016(±0.057.23)	2.986(±0.057.23)	< 3.000
TDS (mg/L)	708(±105.42)	442(±34.12)	1035(±34.12)	1560(±34.12)	2,000

**FAO* Pescod, 1992, FAO 1985, *EC*—Electrical conductivity, *TDS*—Total dissolved solids. The values in parenthesis are the standard deviations

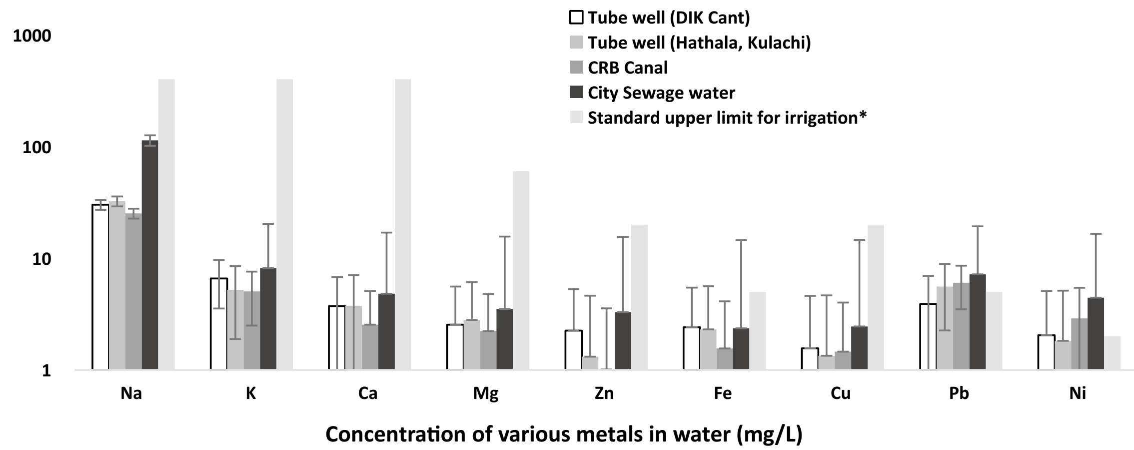

Fig. 2 Concentration of various metals in water used for irrigation and collected from various sources

Table 2 Physical parameters of soil samples treated with water of various sources

Parameters	Soil irrigated with tube well water (DIK Cantonment) ($n=12$)	Soil irrigated with tube well water (Hathala, Kulachi) ($n=12$)	Soil irrigated with CRB canal (Paroa) ($n=12$)	Soil irrigated with sewage water ($n=12$)	WHO limits
Temperature(°C)	15	13.45	13.2	15	N.A
pH	8.6(±1.216)	8.5(±1.145)	7.8(±0.345)	8.7(±1.55)	6.10
EC (dS/m)	0.0745(±0.006848)	0.0437.8(±0.003998)	0.0820(±0.003998)	1.040(±0.003998)	<0.4
Moisture contents (%)	57.3(±7.35)	49.8(±5.10)	60.5(±10.50)	70.9(±11.80)	N.A

(1971). The detailed analysis is provided as supplementary information (ESI).

Digestion of plant materials/samples

Spinach samples were analyzed for the various ions by "Wet digestion" method. A mixture of HNO_3 and $HClO_4$ in the ratio of 9:4 was used for sample digestion, known as di-acid digestion (Pequerul and Perez 1993). A weighed amount of powdered spinach was taken for analysis, poured into china dish and required amount of (1:10) acid mixture was added into it and then the contents were mixed by swirling. The suspension / mixture was placed on a hot plate in the fume hood and heated initially at 80–90 °C and then from 150 to 200 °C. The heating was continued until the stoppage of red NO_2 fumes. The contents were further heated until the volume was reduced to about one third of initial volume and the solution became colorless but not dried. The contents were then cooled and shifted to volumetric flask. The volume was made up to the mark with distilled water and filtered through Whatman filter paper #42 (Motsara and Roy 2008).

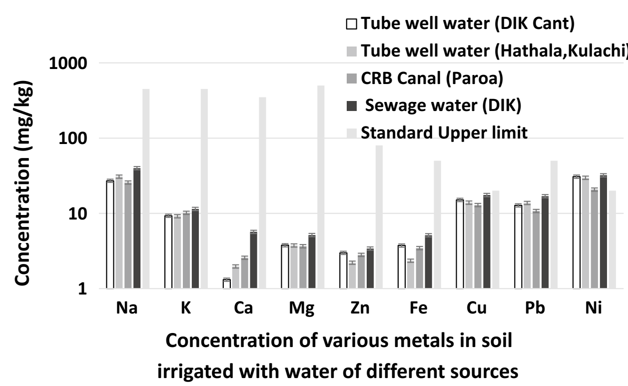

Fig. 3 Concentration of various metals in soil irrigated with different water sources

Quality assurance/quality control

All plastic and glass ware used were soaked previously in HNO_3 (10%) for 24 h. Milli-*Q* water was used to thoroughly rinse the plastic and glass ware. Stock solution of 1000 ppm was diluted to prepare calibration solutions. Working standard solution was prepared by diluting the standard stock solution. Procedural blanks were prepared for quality control along extraction procedure. Correction of the result of each batch (10 samples) was based on the concentration of procedural blank (average $n=3$). Batch of real sample was also prepared correspondingly. Replicate, reagent blanks makeup 20% and 10% each of sample population. Yttium and indium were employed as recovery standard, exhibited 98% and 103% recoveries. After every 10 samples, calibration standard (0.1 ppm) was run to check the stability and sensitivity of instrument. Throughout the experiment, metal's intensity was steady RSD ≤ 6% suggested the stability and sensitivity of instrument.

The concentration of the required elements (Na, K, Ca, Mg, Zn, Fe, Cu, Pb, Ni) were determined by using atomic absorption flamephotometer (Varian AA-240-FS) after properly calibrating the equipment with standard solutions (Motsara and Roy 2008).

Results and discussion

Physical parameters

Water analysis

The results obtained for various physical parameters (pH, EC and TDS) are listed in Table 1. According to Bauder et al. (2010) pH and alkalinity are valuable characteristics that can greatly manipulate the suitability of water for irrigation purposes. The normal pH ranges for irrigation water is 6.5–8.4. But Sheinberg and Oster (1985) reported that the pH of irrigation water is not an accepted criterion of water quality because it tends to be buffered by the soil and most crops can tolerate wide range of pH. The pH of all the water samples were within the permissible limits described by FAO (1985); FAO (1992) except the pH of sewage water (Dappawala band area). The electrical conductivity of tube well Cantonment, Hathala and CRBC, (Paroa) was 1.65(±0.1203) and 1.02(±0.05723), 2.02(±0.5723) dS/m, respectively, and that of waste water was 2.99(±0.057.23) dS/m, Ayers and Westcot (1984) suggested that water having EC higher than 3dS m^{-1} (0.3000 dS/cm) are not safe for irrigational purpose and also according to FAO the standard limits of EC for irrigation water is 3dS m^{-1}. So the EC of water obtained from various sources were suitable for irrigation purpose and within the safe limits. Similarly, the total dissolved solids (TDS) of the water samples were 708(±105.42) NTU in Cantonment water, 442(±34.12) NTU in Hathala water and 1035(±34.12) NTU in CRBC water and 1560(±34.12) NTU for waste water. According to Park et al. (2014), Frenkel (1984), the quality of water used for irrigation is characterized by the color, turbidity, total dissolved solids (TDS), pH, specific conductance, odor, and foam. Colorless, odorless, foamless water with minimum turbidity, TDS below 1000 mg L^{-1} at circumneutral pH and specific conductance below 1.5 m mhos/m, is generally considered to be of good quality for irrigation purposes.

Concentration of metals in water used for irrigation

The results for water obtained from various sources indicated certain differences in terms of metal concentrations in the water samples (Fig. 2). Following trend was observed for concentration of metals in water, sewage water > tube well of DIK Cantonment > Hathala water > CRBC water. It concludes that CRBC water was the best among all. If we compare the contents of various metals then sodium was in highest and zinc was in lowest concentration. However, it is good to know that water collected from all the sources had the metal contents below the permissible limit for irrigation except few like copper and in some cases Ca.

Soil analysis

Physical parameters

The results obtained for physical parameters of soil are reported in Table 2. The mean value of pH of soil irrigated with tube well water Cantonment was 8.6(±1.216) while that of Hathala water 8.5(±1.145), CRBC 7.8(±0.345) and for sewage water was 8.7(±1.55). The EC for the soil irrigated with tube well Cantonment was 0.0745 (±0.006848) dS/m and soil irrigated with tube well Hathala water was 0.04378(±0.003998) dS/m. The EC of the soil irrigated with CRBC and sewage water was 0.0820(±0.0039.98) and 1.04(±0.003998) dS/m, respectively. The results revealed that all the fields had no salinity problem and were within the permissible limits. Similarly, the moisture contents in soil irrigated with tube well Cantonment, Tube well Hathala,

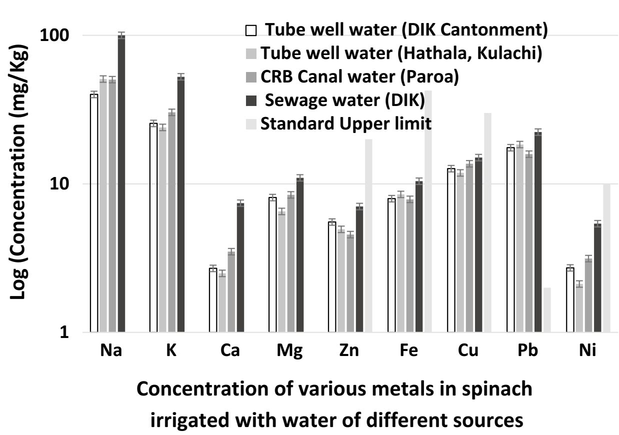

Fig. 4 Concentration of metal contents in spinach irrigated with water obtained from different sources

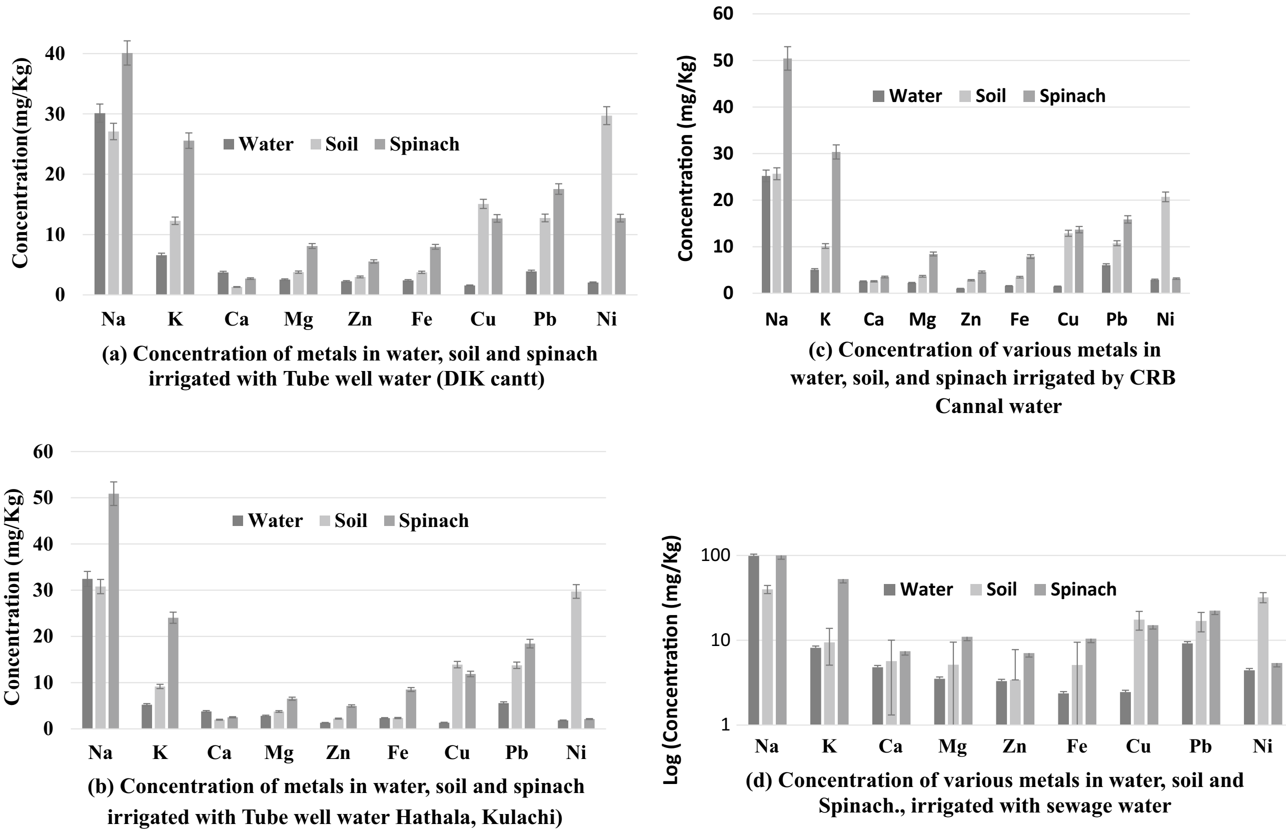

Fig. 5 **a**, **b**, **c**, **d** Concentration of metals in irrigation water, soil and spinach

CRBC, and sewage water were 57.3%(±7.35), 49.8%(±5.10, 60.5%(±10.50), and 70.9%(±11.80), respectively.

Concentration of metals in soil irrigated with various sources of water

The metals concentration detected from soil samples collected from agricultural areas irrigated with various water sources is summarized in Fig. 3. The results revealed higher level of metal fractions in soil samples collected from the land irrigated with sewage water as compared to others. This soil (irrigated with sewage water) shows highest values of every metal ions as compared to others. Due to high concentration of metals, it has greater absorbing/accumulating properties and can be considered as good soil for agriculture purpose. The metal concentration in the soil irrigated by Tube well water Cantonment is second one in most of the cases (except Na, K, and Ca). At third level is the soil irrigated by Hathala tube well water. The soil having least metal contents is the one irrigated by CRB canal water. It is to be noted that none of the metal was above permissible limit except in some cases like copper (Cu allowable limit by WHO is 36 mg/kg) (WHO Switzerland: 1996). It can be further noted that the accumulation of metal ions in soil is almost proportional to contents of these ions in water used for irrigation. This is considered to be one of the major reason of salting of soil. When a soil is irrigated with water having high contents of salt, the soil get deteriorated with the passage of time Awashthi SK (2000); Vinogradov AP (1959).

Plant analysis

Concentration of metals in spinach of different sites

Spinach irrigated with tube well Cantonment, Hathala tube well, CRBC and sewage water was having moisture content as 84.3(±14.13), 83.8(±12.45), 85.2(±13.55), and 86.7(±14.85) %, respectively. Concentration of metals contaminants in spinach irrigated with various sources of water are listed in Fig. 4. It can be noted that all the metals are significantly high in spinach irrigated with sewage water. After sewage water, concentration of metals in most of the cases is high in spinach irrigated by CRB Canal water. (Jan et al. 2009, 2010; Khan et al. 2010). Liu et al. 2005; Ihsanullah et al. 2011; Perveen et al. 2012; Naz et al. 2018; Lone et al. 2003; Mahmood et al. 2020; Flynn (1999). It is to be noted that none of the metal contents were beyond the permissible

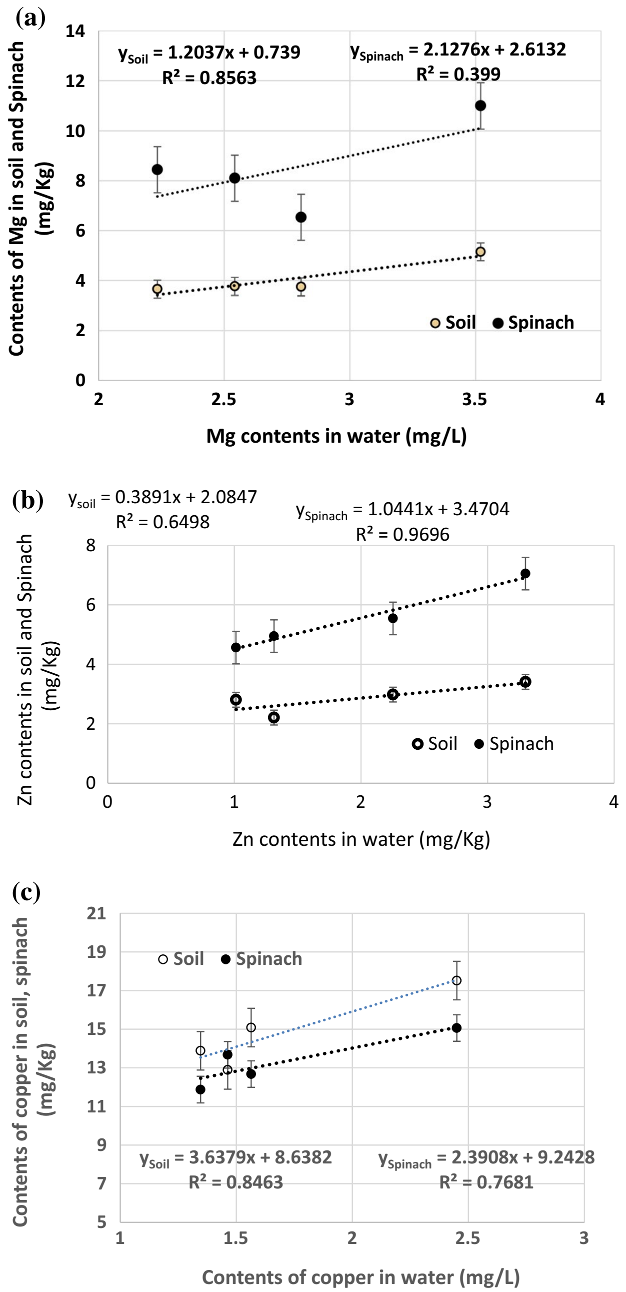

Fig. 6 **a**, **b**, **c** Some of the representative graphs showing the dependence of metal contents in soil and spinach over the metal contents in water

limit. When we look at the metals concentration in spinach it can be noted that the variation of metal is as Na > K > Pb > Cu > Fe > Mg > Zn > Ca > Ni.

To see the impact of water and soil quality over the quality of spinach, we have plotted the metal contents in water, soil and in spinach together in the following Figs. 5a, b, c, d.

It can be noted that contents of various metals in water has the great impact over the contents of soil and spinach. For example sodium contents are high in tube well Cantonment

Table 3 Rate dependence of metal contents in soil and spinach over metal contents in water

Metal	Soil		Spinach	
	Rate	R^2	Rate	R^2
Na	0.176	0.929	0.757	0.959
K	1.732	0.282	4.933	0.592
Ca	0.800	0.328	1.112	0.388
Mg	1.208	0.856	2.128	0.400
Zn	0.389	0.650	1.044	0.970
Fe	0.496	0.307	1.300	0.198
Cu	3.638	0.846	2.391	0.768
Pb	0.833	0.522	0.966	0.605
Ni	0.723	0.027	0.123	0.022

water and same is case in soil and in spinach. On the other hand Ca and Mg contents are low in water same is the case in soil as well as in spinach. Similar trend is observed in case of water obtained from Hathala tube well and CRB canal water with the exception of few cases. Exactly the similar trend was noted in case of sewage water even though the metal contents were high in water. Therefore, it is concluded that water is the main culprit for contamination of soil and plants grown in that soil.

For quantitative relationship we have plotted metal contents in soil and spinach against metal contents in water (Fig. 6a, b). It can be noted that we get a clear dependent of metal contents in spinach and soil over the metal contents in water. Similar trend was observed in all cases except for few metals like Ni. The rate of dependence of metal contents in soil and spinach over metal contents in water is provided in Table 3. In addition, correlation coefficient (R^2) of such dependence is also listed in the same table. If we compare the rate of intake of metal by soil and Spinach, it can be noted that Spinach rate is significantly higher than soil, irrespective of metal except Cu and Ni which are other way round. However, the rate varies from metal to metal (K > Cu > Mg > Fe > Ca > Zn > Pb > Na > Ni). On the other hand soil rate is like Cu > K > Mg > Pb > Ca > Ni > Fe > Zn > Na. whereas, the R^2 values are mostly higher than 0.5 which means the rate are real and not by chance. Therefore, it can be concluded that contents of heavy metals or other depend upon the quality of water. The other factors which control is the texture of soil and type of plant/ vegetable grown over there.

Conclusion

This study contributes detailed understanding of current scenario of contamination of vegetable. The physicochemical parameters are evaluated for water, soil, and spinach samples. The moisture content, TDS and EC values are observed to be in permissible limit of FAO/WHO. A substantial variation in metals accumulation is witnessed in spinach leaves being irrigated with sewage water, followed by canal water and minimum values were recorded for tube well irrigated sites. All the metal contents are within the permissible limits. It is concluded that spinach irrigated by sewage water is more prone to metals as compared to spinach irrigated by canal and tube well water. Although spinach grown on sewage irrigated soil is healthy and contains greater nutrient content than other sources, but it has a negative effect on human health, as sewage water is the main source of heavy metals and other poisonous materials. The metal contents in Spinach and soil were correlated with contents in water used for irrigation and find to have positive correlation. The rate of dependence of metal contents in soil and spinach over water contents varied from soil to soil indicating that both contents in water and soil play a significant role in accumulating the metal in spinach. Strict monitoring of sewage water at the study area should be done. There is an urgent need for the development of strategies to prevent the accumulation of heavy metals in the vegetables and soil.so that the chronic health risk of the population consuming these vegetables can be reduced.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s13762-023-04957-y.

Declarations

Conflict of interest There is no conflict of result between the authors.

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