Eurasian Journal of Soil Science 2024, 13(2), 145 - 152

Eurasian Journal of Soil Science

Journal homepage : http://ejss.fesss.org

Impacts of irrigation with Cd-contaminated water from Sugovushan Reservoir, Azerbaijan on total cadmium and its fractions in soils with varied textures

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Article Info

Received : 11.09.2023

Accepted : 15.01.2024

Available online : 23.01.2024

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Abstract

Cadmium (Cd) presents a significant environmental threat due to its toxic nature and propensity to accumulate in various organs, posing serious health risks upon human exposure. This study focuses on the Sugovushan reservoir in Azerbaijan, aiming to comprehensively understand Cd behavior in soils subjected to varying water levels, shedding light on the intricate interplay between water quality and soil Cd content. Soil samples with distinct textures were collected from a agricultural area in Azerbaijan and subjected to an incubation experiment. The experiment, conducted at 20±0.5°C for 10 days, involved four water levels (%100, %75, %50, and %25 of field capacity) in a randomized complete block design. Cd-contaminated water from Sugovushan reservoir was applied, and inorganic Cd fractions were determined after incubation. The sequential extraction method, as per Shuman's procedure, was employed to assess Cd distribution in exchangeable (EX-Cd), organic (OM-Cd), Mn oxide (MnO-Cd), amorphous Fe oxide (AFeO-Cd), and crystalline Fe oxide (CFeO-Cd) fractions. The soils exhibited varying textures (Sandy Clay Loam, Silty Loam, and Clay) with alkaline reactions, differing salinity, and low organic matter content. Despite somewhat elevated total Cd levels (1.75–2.66 mg/kg), the soils remained below the 3 mg/kg contamination threshold. Water from Sugovushan reservoir, though alkaline, contained Cd concentrations exceeding agricultural use limits. Incubation with Cd-contaminated water increased total Cd content in all soils, with SaCL exhibiting the highest susceptibility. Notably, the SaCL soil showed a significant increase in the exchangeable Cd fraction, emphasizing its environmental risk. This study underscores the importance of soil texture in influencing Cd mobility, especially in low-clay-content soils. The heightened susceptibility observed in SaCL soil highlights the potential threat to food safety, emphasizing the need for sustainable agricultural practices and water management.

Keywords: Cadmium fractions, soil contamination, water quality, environmental risk.

Introduction

Cadmium (Cd) stands out as a particularly hazardous and mobile element in the environment due to its toxic nature and remarkable ability to substitute for calcium in minerals (Thornton, 1986; Alloway and Jackson, 1991; Nies, 1999, 2003). The repercussions of Cd contamination extend beyond its environmental presence, as it tends to accumulate in various organs upon entry into the human body, posing serious health risks (Pan et al., 2010; Hajeb et al., 2014). Unlike some other toxic elements, such as mercury (Hg) and arsenic (As), Cd predominantly finds its way into the human diet through terrestrial pathways, with vegetables grown in regions characterized by elevated Cd concentrations in soil and groundwater being primary contributors (Sebastian and Prasad, 2014; Liu et al., 2017; Tefera et al., 2018). The nuanced bioavailability of Cd becomes

doi : https://doi.org/10.18393/ejss.1424421

: http://ejss.fesss.org/10.18393/ejss.1424421

Publisher : Federation of Eurasian Soil Science Societies

e-ISSN : 2147-4249

apparent in regional variations, exemplified by the differing Cd levels in rice cultivated in the southern and northern parts of China. These variations can be attributed to factors such as soil acidity, nitrogen fertilizer use, pollution through irrigation, and crop selection (Chen et al., 2018).

Recognizing the significant threats posed by Cd to both human health and the environment, regulatory frameworks like the European Water Framework Directive and the European Groundwater Directive have implemented management plans aimed at mitigating Cd releases into the environment (EC, 2000, 2006). Various countries have responded by establishing threshold values for Cd concentrations in groundwater and drinking water, underscoring the necessity for robust monitoring and control measures (UNEP, 2010; WHO, 2011). While numerous studies have delved into the behavior of Cd in soils and groundwater, exploring its agricultural impact, bioavailability, and environmental remediation (Carrillo-González et al., 2006; Bigalke et al., 2017), many of these efforts have been compartmentalized, focusing on specific issues or localities. This has resulted in a fragmented understanding of Cd dynamics in diverse environments (Akbar et al., 2006; Karak et al., 2015).

This study aims to contribute to the comprehensive understanding of Cd behavior in the environment, specifically focusing on the Sugovushan reservoir in Azerbaijan. By investigating the changes in Cd concentrations and fractions in soils subjected to different levels of water from the Sugovushan reservoir, we aim to shed light on the intricate interplay between water quality and soil Cd content.

Material and Methods

Soil sampling, preparation and analysis

The soil samples with three distinct textures, intended for use in the incubation experiment, were collected from agricultural area in Azerbaijan, collected from a depth of 0-20 cm. The collected soil samples underwent meticulous cleaning to remove stones and plant residues from the soil surface. Subsequently, these soil samples were transported to the laboratory for further analysis. In the controlled laboratory conditions, the soil samples were subjected to a series of procedures. Initially, the samples were air-dried in a cool and shaded environment to prevent alterations in their chemical composition due to excessive heat or sunlight. Once dried, the soil samples were finely ground after eliminating any remaining moisture. This grinding process facilitated homogenization, ensuring uniformity for subsequent analyses. The soil samples were then sieved through a 2 mm mesh to achieve a consistent particle size, optimizing the analytical results. The prepared soil samples, now in a homogeneous and fine-grained state, were considered analytically ready and were utilized for subsequent investigations. Various parameters were determined in the conducted soil analyses using established scientific methods. The soil texture was determined using the hydrometer method as described by Bouyoucos (1962). pH and Electrical Conductivity (EC) were measured in a 1:1 (w/v) soil-to-distilled water suspension using a pH meter and EC meter (Peech, 1965; Bower and Wilcox, 1965). Organic matter content was assessed through wet oxidation with $K_2Cr_2O_7$, following the method proposed by Walkley and Black (1934). Calcium carbonate ($CaCO_3$) content was determined volumetrically using the Scheibler calimeter (Rowell, 2010). The water capacity (field capacity, wilting point, and available water) was determined as reported by Klute (1965) and Peters (1965). Additionally, available heavy metals (Fe, Cu, Zn, Mn, Cd, Pb, Ni) were determined using the DTPA extraction method, total heavy metals determined using the aqua regia + HF digestion method followed by analysis with Atomic Absorption Spectrophotometry (Lindsay and Norvell, 1978, EN 13656, 2002).

Water sampling, preparation and analysis

Water samples, including those from Sugovushan reservoir (40.323985 N, 46.743843 E) in Azerbaijan, were collected for analysis. The water samples were promptly transported to the laboratory and filtered using Whatman No. 41 filter paper. pH and electrical conductivity analyses were conducted using a pH meter and an EC meter, respectively. The analysis of anions (Cl^-, HCO_3^-, SO_4^{2-}, NO_2^-, NO_3^-, PO_4^{3-}.) and cations (Ca^{2+}, Mg^{2+}, Na^++K^+, NH_4^+) in the water followed the methods outlined by the US Salinity Laboratory Staff (1954). The contents of heavy metals (Fe, Cu, Zn, Mn, Cd, Pb, Ni) in the water were determined using an Atomic Absorption Spectrophotometer.

Soil incubation experiment

The experiment was conducted in a constant temperature incubator at 20±0.5°C for a duration of 10 days. The field capacity, wilting point, and available water content of the soils used in the experiment were determined according to the methodology reported by Klute (1965) and Peters (1965). The incubation experiment was established in a randomized complete block design with four different water levels (%100, %75, %50, and %25 of field capacity) and three replications. For this purpose, 50 g of each soil sample was measured and placed into a 100 mL plastic beaker. Plant-available water from Sugovushan reservoir, enriched with Cd, was then added to the soil samples at the four different water levels mentioned above. After thorough mixing, all samples were covered with a piece of parafilm containing pores to facilitate air influx while preventing the evaporation of soil water. The samples were stored in the dark at a constant temperature of 20°C throughout the incubation period. On the 10th day of incubation, soil samples were collected, and the inorganic Cd fractions were determined.

Cadmium Fractionation

Total soil Cd was determined using aqua regia + HF digestion method (Shuman, 1979). Cadmium distribution in the exchangeable (EX-Cd), organic (OM-Cd), Mn oxide (MnO-Cd), amorphous Fe oxide (AFeO-Cd) and crystalline Fe oxide fractions (CFeO-Cd) were deter-mined according to Shuman method. The solids remaining were analyzed by complete dissolution in inorganic acids (HCl–HNO_3 and HF) and the fraction designed residual (Res.-Cd) (Shuman, 1979; 1983; 1988). Cd contents of the all fractions and total Cd contents in the filtered solution was determined by atomic absorption spectrophotometry. The general procedures of the sequential extractions are given in Table 1.

Table 1. Cd fractionation procedure

Fraction	Solution	Soil, g	Solution, ml	Conditions
Exchangeable (EX-Cd)	1M $Mg(NO_3)_2$ (pH 7)	10	40	Shake 2h
Organically complexed (OM-Cd)	0.7M NaOCl (pH 8.5)	10	20	30 min in boling water bath. Stir occasionally. Repeat extraction
Manganese oxide bound (MnO-Cd)	0.1M $NH_2OH.HCl$ (pH 2)	1*	50	Shake 30 min
Amorphous iron oxide bound (AFeO-Cd)	0.2M $(NH_4)_2C_2O_4$ in 0.2 M $H_2C_2O_2$ (pH 3)	1	50	Shake 4h in the dark
Crystalline iron oxide bound (CFeO-Cd)	0.1M ascorbic acid in the above oxalate solution	1	50	30 min in boling water bath. Stir occasionally

*One gram from step 2 that is dried, ground and passed through a 0.5 mm screen

Results

The characteristics of the soils used in the incubation experiment are presented in Table 2. According to the obtained results, the soils selected for the experiment exhibit differences in terms of texture. Specifically, one of the experimental soils is classified as 'Sandy Clay Loam,' another as 'Silty Loam,' and the third as 'Clay.' All soils have an alkaline reaction and are calcareous. While the SaCL soil is non-saline, the others are saline. Additionally, the organic matter content in all soils is observed to be low. According to the analysis conducted by Kloke (1980), no heavy metal pollution is detected in the soils used for the experiment, and the heavy metal contents of the soils do not exceed their buffering capacity. Nevertheless, the total Cd contents of soils exhibiting different textural characteristics used in the incubation experiment were determined as 1.75, 2.12, and 2.66 mg/kg, respectively. It has been documented that soil texture, particularly the increase in clay and organic matter content, is associated with higher mean Cd concentrations in soils (Holmgren et al., 1993). The threshold value for considering soil as contaminated with Cd is generally set at concentrations above 3 mg/kg (Akbar et al., 2006). Concentration gradients are frequently observed in proximity to industrial installations, roads, and urban areas (Page et al., 1987; Joimel et al., 2016). Therefore, it can be asserted that the soils used in the experiment do not exhibit significant contamination with Cd. Since the soils for the experiment were sourced from agricultural areas in Azerbaijan, where phosphorus-containing chemical fertilizers are commonly used to enhance rice yields (Islamzade et al., 2024), the total Cd content of the soil, while somewhat elevated, remains below the threshold of 3 mg kg^{-1}, indicating an acceptable level of non-contamination.

The chemical properties of the water used in the incubation experiment are presented in Table 3. The Sugovushan Reservoir is fed by the Terter River. The River Terter is the largest river in the Karabakh region in Azerbaijan, serving the agricultural and domestic needs of over 400 thousand inhabitants in the surrounding area for many years. Unfortunately, between 1994 and 2020, the Terter River, which was occupied by Armenian forces, witnessed extensive contamination from numerous mining sites, with gold mining being the most detrimental. The lack of any legal norms for environmental protection in Karabakh during the occupation allowed mining operators to dispose of all their waste into the river. As a result, not only did the ecology suffer, but also the Terter River and the Sugovushan Reservoir on this river became polluted, primarily with Cd. This water sample demonstrates an alkaline reaction and contains some heavy metals within its composition. Among these heavy metals, the concentration of Cd exceeds the recommended upper limit for agricultural use of water, set at 0.01 mg L^{-1} (FAO, 1985), measuring at 1.64 mg L^{-1}. However, for other heavy metals, there is not a significantly increased risk similar to that posed by Cd.

Table 2. Characteristics of the soils used in the incubation experiment

		SaCL	SiL	C
	Sampling point	38.6322740 N 48.8646310 E	40.2559770 N 47.6289990 E	40.5438710 N 47.2880790 E
Texture	Sand, %	50,69	11,55	6,76
	Silt, %	15,91	78,58	7,69
	Clay, %	33,41	9,87	85,55
	Class	Sandy Clay Loam	Silty Loam	Clay
Soil water properties	Field Capacity, % Vol	32,30	30,90	44,90
	Wilting point, % Vol	21,60	10,40	35,00
	Available Water, % Vol	10,70	20,50	9,90
	Bulk density, g cm^{-3}	1,48	1,37	1,19
Chemical properties	pH	7,70	8,17	7,86
	EC, dSm^{-1}	0,51	7,62	4,77
	$CaCO_3$, %	12,93	15,06	6,69
	Organic matter, %	1,61	0,88	2,47
Available heavy metals	Fe, mg kg^{-1}	65,50	6,21	35,71
	Cu, mg kg^{-1}	7,54	1,80	7,74
	Zn, mg kg^{-1}	0,58	0,31	0,43
	Mn, mg kg^{-1}	23,01	3,76	7,34
	Cd, mg kg^{-1}	0,20	0,16	0,15
	Pb, mg kg^{-1}	2,48	3,58	3,25
	Ni, mg kg^{-1}	3,68	2,15	3,59
Total heavy metals	Fe, %	3,12	3,81	5,39
	Cu, mg kg^{-1}	84,82	75,36	95,15
	Zn, mg kg^{-1}	191,17	185,58	296,61
	Mn, mg kg^{-1}	0,18	0,13	0,25
	Cd, mg kg^{-1}	1,75	2,12	2,66
	Pb, mg kg^{-1}	86,85	93,19	95,85
	Ni, mg kg^{-1}	75,69	65,48	86,15

Cd-contaminated water from the Sugovushan reservoir was applied to soils of three different textures (SaCL, SiL, and C) in this experiment, and changes in the soil's total and Cd fractions were assessed after irrigation with 100%, 75%, 50%, and 25% of the available water content. Figure 1 illustrates these variations, including the initial Cd fractions of the soils before irrigation. According to the obtained results, all soils irrigated with Cd-contaminated water exhibited an increase in their total Cd content. This increase was found to be correlated with the amount of water applied to the soil. In effective agricultural irrigation, it is desirable for soil moisture levels to be at field capacity (Kumar et al., 2023). Initially, the Cd levels in SaCL, SiL, and C soils were 1.75, 2.12, and 2.66 mg kg^{-1}, respectively. However, when soils were irrigated with 100% of the available water content, the total Cd contents increased to 2.10, 2.79, and 2.98 mg Cd kg^{-1} for SaCL, SiL, and C soils, respectively. A significant decrease in total Cd content was observed in all soils when the amount of applied water decreased. Similarly, previous studies have reported a significant increase in Cd and other heavy metal contents in soils irrigated with Cd-contaminated water (Chaoua et al., 2019; Orosun et al., 2023; Shahriar et al., 2023).

Table 3. Chemical properties of the water used in the incubation experiment

pH	8,00	**Anions**		**Heavy metals**	
EC, dSm^{-1}	3,10	Cl^-, mg L^{-1}	20,40	Fe, mg L^{-1}	210,00
Cations		HCO_3^-, mg L^{-1}	114,60	Cu, mg L^{-1}	<0,01
Ca^{2+}, mg L^{-1}	38,1	SO_4^{2-}, mg L^{-1}	67,90	Zn, mg L^{-1}	<0,01
Mg^{2+}, mg L^{-1}	9,90	NO_2^-, mg L^{-1}	0,01	Mn, mg L^{-1}	4,17
Na^++K^+, mg L^{-1}	64,80	NO_3^-, mg L^{-1}	1,43	Cd, mg L^{-1}	1,64
NH_4^+, mg L^{-1}	0,15	PO_4^{3}, mg L^{-1}	0,10	Pb, mg L^{-1}	9,82
Total cations	112,95	Total anions	204,44	Ni, mg L^{-1}	<0,01

In the incubation experiment, although initially the entire set of soils used exhibited the CFeO-Cd fraction as the predominant fraction within inorganic Cd fractions, it was observed that OM-Cd and Res.-Cd contained the least Cd fraction (Figure 1). Numerous studies have demonstrated the influence of various soil physicochemical properties such as soil texture, organic matter content, and pH on the distribution of inorganic Cd fractions in soils (Anju and Banerjee, 2011; Nejad et al., 2021; Lian et al., 2022). Kızılkaya and Aşkın (2002), in their investigation of the distribution of Cd fractions and the relationships between soil properties in agricultural fields in the Bafra Plain of Turkey, determined that the total Cd content in alluvial soils ranged from 1.83 to 2.73 mg kg^{-1}. They found that 7.3-18.5% of total Cd in the soils consisted of EX-Cd, 4.1-10.8% of OM-Cd, 6.1–7.6% of MnO-Cd, 5.2–8.7% of AFeO-Cd, and 5.8–7.2% of CFeO-Cd. Additionally, in this study, significant positive correlations were identified between the distribution of Cd fractions in soils and the clay content and cation exchange capacity of the soils.

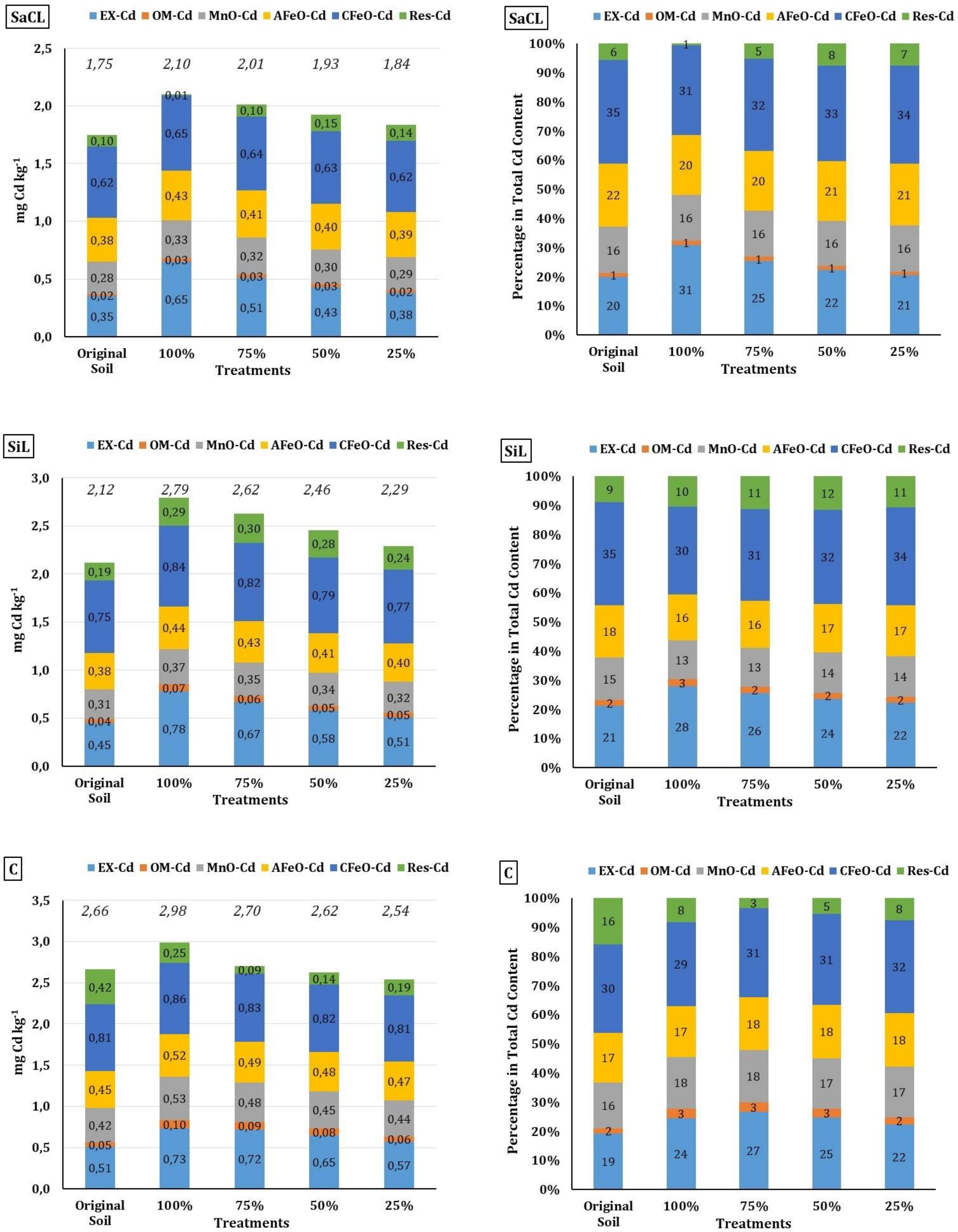

Figure 1. Changes in the different textural (SaCL, SiL and C) soil's total and Cd fractions were assessed after irrigation with 100%, 75%, 50%, and 25% of the available water content

After irrigation with Cd-enriched water in soils of different textures (SaCL, SiL, and C), increases were observed not only in the total Cd content of the soils but also in the Cd fractions. Remarkably, the increase in Cd fractions occurred in the EX-Cd fraction of the SaCL soil (Figure 1). This observation is attributed to the lower clay content of the SaCL soil compared to the other two soils (SiL and C). The exchangeable Cd fraction is particularly crucial in environmental risk assessments and soil quality management due to its susceptibility to plant uptake. This exchangeable fraction (EX-Cd) is closely monitored to assess and regulate the environmental impacts of Cd. Consequently, when low-clay-content soils are used for agricultural purposes in the presence of Cd-contaminated water, it is suggested that Cd can be more readily taken up by plants from the soil, potentially entering the food chain. This underscores the importance of monitoring and managing Cd's exchangeable fraction in mitigating the environmental implications associated with Cd-contaminated waters used for agricultural irrigation.

Conclusion

In conclusion, this study contributes valuable insights into the behavior of Cd in soils, particularly in the context of the Sugovushan reservoir in Azerbaijan. The incubation experiment revealed the nuanced response of soils with different textures to irrigation with Cd-contaminated water, emphasizing the importance of soil physicochemical properties in shaping Cd distribution. The SaCL soil, characterized by lower clay content, exhibited a heightened susceptibility to Cd mobility, particularly in the exchangeable fraction. This underscores the significance of considering soil texture in managing and mitigating the environmental risks associated with Cd-contaminated water used for agricultural purposes.

The findings highlight the potential implications for food safety as Cd may readily enter the food chain when low-clay-content soils are irrigated with contaminated water. Therefore, sustainable agricultural practices and water management strategies need to consider soil characteristics to minimize the risk of Cd exposure. As future research endeavors unfold, a more comprehensive understanding of the intricate relationships between water quality, soil properties, and Cd behavior will be crucial for developing effective strategies to safeguard environmental and human health.

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