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CHEMICAL PROPERTIES AND TOXICITY OF SOILS CONTAMINATED BY APPLICATION OF WASTEWATER SLUDGE OF HIGH HEAVY METALS CONTENT

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Abstract

Heavy metal (HM) contamination of agricultural soils is a worldwide ecological problem. Wastewater sludges (WWS) produced from communal wastewater treatment plants is commonly recognized as a significant basis of nutrient and soil conditioner for cultivating use. Determination of intensities of HMs in WWS is essential prior to application of the sludge to agriculture for of the intrinsic risk of HM toxicity to soil. Wastewater sludge created from different WW sources were analysed for a range of total HMs using ICP-AES. Most of the samples analysed show agreement with respect to the regulatory parameters established by the USEPA, South Africa and EU guidelines for levels of HMs in WWS envisioned for agricultural soil application. In this study, the concentrations of HMs (Cd, Cu, Ni, Pb, and Zn) were investigated in the agricultural soils before and after WWS applications. Wastewater sludge produced from the Hódmezővásarhely WW treatment plant in general has high HMs due to the presence of several industries in the area. The variations in the HM concentrations were due to the application ratio of WWS to the soil. Considering the nutrient and soil conditioning values of the WWS and reusing them for agriculture is an economical and environmentally sound option and deserves given greater attention.

Keywords: *Soil, Heavy metals, Toxicity, wastewater sludge*

1. INTRODUCTION

Soil is a complex amalgam, a non-renewable natural resource because it cannot be re-created except within the context of geological timescales. It can be simply defined as the unconsolidated mineral or organic material on the immediate surface of the Earth that serves as a natural medium for the growth of land plants [1].

Heavy metals (HM) are elements with an atomic density greater than 6 g/cm3; they are one of the most persistent pollutants in wastewater. Heavy metals also known as trace metals, are one of the most persistent pollutants in wastewater. Heavy metals are highly hazardous to the environment and organisms.

It can be enriched through the food chain. The persistence of HMs in WW is due to their non-biodegradable and toxicity nature [2]. Once the soil suffers from HM contamination, it is difficult to be remediated [3].

Heavy metal contamination of agricultural soils is a worldwide ecological problem. Heavy metal contamination of agricultural soils has become a significant environmental problem [4]. This remarkable industrialization has led to the continuous accumulation of HM ions in eco-environment and caused deterioration of many ecosystems and social health. Anthropogenic inputs such as agricultural activities, energy conversion and production, metallurgy and mining, microelectronics, solid and liquid waste disposal have been the major sources of HM ions accumulated in our environment.

Wastewater is a waste product produced at the end of communal and industrial wastewater treatment processes and is being produced in gradually large volumes global due to increasing population and growing urbanization. Wastewater sludge (WWS) originated from industrial estates contains toxic substances such as HM, recalcitrant organics and other undesirable pollutants which may accumulate in the edible parts of food crops and pose serious threats to human life. These are hazardous to water resources, agriculture, ecosystems and the human population [5]. Wastewater sludge contains HM which may be lethal for animals and humans if their concentrations exceed the permissible limits [6]. Wastewater irrigation, solid waste disposal, sludge applications, vehicular exhaust and industrial activities are the most important sources of HM contamination of soil and food crops grown on contaminated soils [7].

The most common toxic HMs in WWS include arsenic (As), lead (Pb), mercury (Hg), cadmium (Cd), chromium (Cr), copper (Cu), nickel (Ni), silver (Ag), and zinc (Zn). The release of high amounts of HMs into water bodies creates serious health and environmental problems and may lead to an upsurge in WW treatment cost.

Some of the negative impacts of HMs on plants include decrease of seed germination and lipid content by Cd, decreased enzyme activity and plant growth by Cr, the inhibition of photosynthesis by Cu and Hg, the reduction of seed germination by Ni and the reduction of chlorophyll production and plant growth by Pb [8]. The impacts on animals include reduced growth and development, cancer, organ damage, nervous system damage and in extreme cases, death.

Soils may become contaminated by the accumulation of HMs and metalloids through emissions from the rapidly expanding industrial areas, mine tailings, disposal of high metal wastes, leaded gasoline and paints, land application of fertilizers, animal manures, sewage sludge, pesticides, WW irrigation, coal combustion residues, spillage of petrochemicals, and atmospheric deposition [9, 10].

The application of WWS on soils has been widespread in agricultural areas. It depends on soil properties, HM levels and characteristics, plant species and climatic conditions. Several studies have been carried out on the impacts of WW irrigation on the accumulation of HM in soil and plant systems around the world [7].

Removal of WWS presents a problem that touches a set of complex concerns including health and environment as well as economics. The European Union Directive (91/271/EEC) for example prohibits removal of sludge to the marine environment [11]. Soil application is simple and economical but soil is becoming scarce.

Wastewater sludge is usually reflected as waste, the recycling and reuse of valuable nutrients contained in the WWS are currently being measured as important resources for sustainable development [12]. Treated WWS can be reprocessed in numerous ways including its use as fertilizer with important nutrient additions improving plant growth and as a soil conditioner for improving the physical and chemical properties of soils [13-15].

The estimated percentages of WWS applied to agricultural soils have been mentioned as 29% in the USA, 40% in the UK, 60% in France, 30% reuse potential in Russia and 230,000 ton/year in Japan [16-19]. In developing countries, the experience of using WWS application for agricultural purposes is often kept to communal farms. There is a supposed risk to the environment that prevented wastewater sludge from existence application to agricultural soils.

In addition to heavy metals and pathogens, other harmful and toxic pollutants such as pharmaceuticals, detergents, various salts, pesticides, toxic organics, flame retardants and hormone disruptors can also be present in wastewater sludge [20-22]. Wastewater sludge can inoculate in soils extreme amount of nutrients, pesticides and can increase soil salinity [23].

Heavy metals are mainly originated in WWS since due to their hydrophobic nature; they are related with the solid portion of the WW [24]. Frequently, communal wastes have lesser HM contents than industrial wastes. As a result, toxic metals such as Pb, Cd, Hg, Ni and Cr may be current present in municipal WW due to heavy urbanization and the entry of raw industrial WW into the municipal WW system [25, 26]. On the basis of relative toxicity to plants and animals, two groups of heavy metals can be recognized. The first group including Cd and Pb are highly toxic to humans and animals on the other hand, they are less toxic to plants. The second group containing Zn, Ni and Cu are, when present in excess concentration, more damaging to plants than to humans and animals [27]. The long-term use of WWS can cause HM accumulation in soils [28]. Even after a short term WWS application, the HMs levels in soils can increase significantly. For example, Oliveira and Mattiazzo [29] observed increases in Cu, Cr, Ni and Zn concentrations in soils amended for 2 years with WWS. Wastewater sludge application rate is an important factor that determines the extent of accumulation of HMs in soils and their absorption by plants. Increased concentrations of Pb, Cd, Ni and Cr have been observed in plant seeds as a result of increased application of sludge [30]. The level of plant uptake, bio-accumulation and tolerance of plants to HMs varies among different crops at different rates of application of WWS [31]. The regulatory limits of HMs in WWS from a number of countries are summarized in Table 1.

Table 1: Regulatory limits of heavy metals in wastewater sludge intended for agricultural application [32-33]

Heavy Metal	EU Directive (86/278/EEC)	Chinese Regulation (GB 18918 - 2002)	USA Regulation (40 CFR Part 503, 503.13)	South African Guideline (Pollutant Class a)	Heavy Metal	Units	EU Directive (86/278 /EEC)	Chinese Regulation (GB 18918-2002)
	Limit value (mg/kg)	Limit value (kg/ha. year)	Acidic soil pH<6.5	Neutral and Basic soil pH>6.5	Pollutant dose Limits (mg/kg)	Annual pollutant loading rate (kg/ha. year)	Pollutant limit mg/Kg (Class a)	Pollutant limit mg/Kg (Class b)
Zn	2500 - 4000	30	500	1000	2800	140	2800	7500
Cu	1000 - 1750	12	250	500	1500	75	1500	4300
Cd	20 - 40	0.15	5	20	39	1.9	40	85
Ni	300 - 400	3	100	200	420	21	420	420
Pb	750- 1200	15	300	1000	300	15	300	840

Additional risk of contamination in the environment is observed through leaching of HMs from soils to ground water. The risk increases with time as metals are persistent in soils over a longer time and many metals do not suffer biochemical degradation thereby increasing the risk of bioavailability of metals and their leaching to ground water table [34].

At present large amounts of WWS are being produced and stored in several WW treatment plant premises in world and agricultural application is being careful as a future practical choice of removal. This research is aimed at assessing the effects of metals such as Cd, Cu, Ni, Pb and Zn and toxicity of soil subjected to strong human pressure associated with pollution.

2. MATERIALS AND METHODS

In a greenhouse study, the soil samples used in pot experiment were clay loam brown forest and chernozem meadow collected from farmland surface layer (0–200 mm) of an agricultural area of Gödöllő and Szeged (Hungary), respectively. Three different WWSs were selected depending on their HMs content. One is characterized as low (LHM) and the second is medium (MHM) HMs content originated from Nyíregyháza wastewater treatment plant and the third sample of high HMs content (HHM) was collected from Hódmezővásárhely wastewater treatment plant. The main physico-chemical parameters of soil and WWSs are shown in Table 2. Fresh soil samples were sieved through a 4 mm sieve and mixed with WWSs to form 15 and 60% (soil : sludge; w/w), and then placed into plastic pots with 42 cm in height and 23 cm in diameter. All treatments were designed in triplicates and submitted for statistical analysis. The study was conducted to determine the adverse effect of HM pollution in the soils amended with communal WWS for 16 weeks.

Table 2: Physico-chemical properties of soils and wastewater sludge samples

Parameters	Origin of Soil Samples		Origin of Wastewater Sludge		
	Gödöllő	Szeged	Nyíregyháza		Hódmezővásárhely
			LHM	MHM	HHM
Soil type	clay loam brown forest	chernozem meadow			
$pH_{(H2O)}$	5.12	6.02	7.99	7.17	7.8
Dry matter, %	22.4	28.6	74	70	42.9
Organic matter, %	1.27	2.87	25.6	48.2	20.4
Humus content, %	1.24	3.55	-	-	-
Salt content, %	0.74	0.81	-	-	-
$CaCO_3$, %	1.01	2	-	-	-
Total N content, mg kg^{-1}	84.11	134.7	75.700	98.900	43311
NO_3-N, mg kg^{-1}	133.08	39	-	-	-
NH_4-N, mg kg^{-1}	410.69	4.5	-	-	-
Ca, mg kg^{-1}	856	443	5707	29724	27333
Mg, mg kg^{-1}	203	257	2810	5072	11860
Na, mg kg^{-1}	21	53	1290	1349	1441
AL-P_2O_5, mg/kg	121.31	378	9700	9100	20104
AL-K_2O, mg/kg	107	428	3120	3596	2908
Zn, mg kg^{-1}	38.1	1.1	453	634	1068
Cu, mg kg^{-1}	22.9	2.4	100	161	182.3
Cd, mg kg^{-1}	0.18	1.02	1	2.4	4.168
Ni, mg kg^{-1}	0.064	0.077	15	39.2	56.9
Pb, mg kg^{-1}	15.1	0.96	30	83	540.7

AL: Ammonium lactate soluble P and K

Soil samples were analysed for physico-chemical properties. Total carbon content was measured by using a dry combustion method [35]. Total organic matter (OM) content was calculated by multiplying the total C values by 1.72 [36]. The pH of the soil suspension with soil : water at the ratio of 1:5 was measured by using a pH meter. Heavy metals in soils were fractionated by a modified procedure of Amacher [37]. The parameters include: pH, moisture content, nitrogen (N), phosphorous (P) and potassium (K).

The total heavy metal determination was done using two methods for comparison and quality assurance purposes. The methods used were atomic absorption spectrometer (Varian–AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). The heavy metals analysed include: Cd, Cu, Pb, Ni, and Zn.

Total organic carbon (TOC) was analyzed by dichromate ($K_2Cr_2O_7$) oxidation and titration with ferrous ammonium sulphate [38]. The water soluble C (WSC) contents of the liquid fractions were determined spectrophotometrically at 590 nm after addition of $K_2Cr_2O_7$ and H_2SO_4 (digestion at 150°C for 15 min) according to Sims and Haby's [39] method. The hot-water extractable C (HWEC) fraction of SOM can be determined quickly by simple analytical method. The HW extraction technique delivers a fractionation according to turnover rates of SOM by keeping a soil : water mixture (1:5 w/v) for 60 min under reflex [40].

3. RESULTS AND DISCUSSION

Investigation of HMs fractions in soils amended with WWS would ascertain their availability and contamination level in soils. This study investigated HM fractions in soils after 8 weeks incubation in the greenhouse at 25+2°C with moisture content 40%.

The ranges of HM concentrations in soils amended with WWS were apparently wide. All fractions were significantly higher in the soil samples amended with HHM content WWS of Hódmezővásárhely than the samples treated with Nyíregyháza WWS. Heavy metal concentrations varied in the soils as Pb > Cu > Ni > Zn > Cd after WWS amendment.

In LHM and MHM concentrations differed in soils as Zn > Pb > Cd > Cu > Ni. Repeated WWS treatments increased pH values of the soils. The higher EC of soil indicated an accumulation of salts in the soils due to WWS amendment. The results of analysis of the physico-chemical characteristics of the soil : sludge mixture are summarized in Table 3.

Summaries of the results of the ICP-AES analysis of HMs in the WWS samples collected from the two WW treatment plants are given in Table 3A, 3B and 3C.

The results showed that HM fractions were significantly ($P < 0.05$) higher in soils amended with HHM-WWC of Hódmezővásárhely as compared to the soils samples treated with WWS originated from Nyíregyháza.

In general, most of the HMs are present in low concentrations and below the regulatory limits provided by the USEPA, South Africa and the European Union. These concentrations of Cu are generally low and well below the USEPA, South African and EU guidelines. This indicates that the sludge samples carry lower risk with respect to Cu toxicity.

Table 3: Physico-chemical characteristics and heavy metals content in soil samples and incubated for 16 weeks with different application ratios of wastewater sludge samples with different heavy metals contents

Parameter	A. Soil samples amended with LHM-WWS			
	15%		60%	
	Gödöllő	Szeged	Gödöllő	Szeged
pH	4.8	6.14	5.07	6.29
Moisture content, %	12	13.5	18.6	25.1
Organic carbon, %	1.47	3.13	1.96	3.87
Organic matter, %	2.53	5.38	3.37	6.66
Nitrogen, %	90.04	137.18	99.12	160.17
AL-P_2O_5, mg/kg	143	432	171	690
AL-K_2O, mg/kg	121	488	143	601
Zn, mg kg^{-1}	40.7	2.01	48.9	4.1
Cu, mg kg^{-1}	26.4	3.01	35.2	3.6
Cd, mg kg^{-1}	0.41	1.12	0.67	2.76
Ni, mg kg^{-1}	0.07	0.08	0.09	0.11
Pb, mg kg^{-1}	17.9	1.99	24.2	4.76
Parameter	B. Soil samples amended with MHM-WWS			
	15%		60%	
	Gödöllő	Szeged	Gödöllő	Szeged
pH	4.95	6.38	5.34	6.57
Moisture content, %	13.6	14.4	19.6	26.4
Organic carbon, %	1.55	3.46	2.39	4.15
Organic matter, %	2.67	5.95	4.11	7.14
Nitrogen, %	99.7	144.5	103.72	179.7
AL-P_2O_5, mg/kg	155	513	185	725
AL-K_2O, mg/kg	133	497	161	684
Zn, mg kg^{-1}	48.1	3.44	53.7	5.2
Cu, mg kg^{-1}	34.7	5.08	41.5	4.8
Cd, mg kg^{-1}	0.55	1.87	0.95	3.17
Ni, mg kg^{-1}	0.08	0.09	0.11	0.16
Pb, mg kg^{-1}	19.4	2.59	33.2	5.96
Parameter	C. Soil samples amended with HHM-WWS			
	15%		60%	
	Gödöllő	Szeged	Gödöllő	Szeged
pH	5.16	6.61	5.66	6.82
Moisture content, %	15.5	16.7	21.6	30.1
Organic carbon, %	1.67	3.78	2.95	4.99
Organic matter, %	2.87	6.50	5.07	8.58
Nitrogen, %	112.2	164.3	133.8	201.1
AL-P_2O_5, mg/kg	165	572	192	767
AL-K_2O, mg/kg	145	512	176	714
Zn, mg kg^{-1}	53.3	5.76	61.2	6.6
Cu, mg kg^{-1}	45.4	5.77	49.1	5.5
Cd, mg kg^{-1}	0.64	2.04	1.32	3.78
Ni, mg kg^{-1}	0.09	0.109	0.14	0.19
Pb, mg kg^{-1}	21.1	2.99	38.8	6.49

The Chinese guidelines are exceeded but the limiting concentrations given by Chinese regulations are generally low.

Copper is among the trace elements that are essential to life although at high concentrations Cu is toxic. Copper may be derived from cleaning products, cosmetics and shampoos, fuels, inks, medicines and ointments, food products, oils and lubricants, paints and pigments, polish and wood preservatives, electronics, plating, paper, textile, rubber, fungicides, printing, plastic, and brass and other alloy industries and it can also be emitted from various small commercial activities and warehouses, as well as buildings with commercial heating systems.

The Ni concentrations are mostly below the Chinese and South African guidelines. These results also indicate that the sludge samples carry very low risk with respect to Ni toxicity. Nickel is toxic to humans and both can reach the food chain via plant uptake from contaminated soil [37]. The Pb concentrations as are low and well below all the regulatory guidelines considered. Lead is known for its immobility and limited translocation in plants.

The results indicate once again the low risk of the WWS samples collected from the two treatment plants with respect to lead toxicity on the basis of total Pb metal concentrations. Lead is defined by the United States Environmental Protection Agency (USEPA) as potentially toxic to most forms of life [41]. A major source of Pb in WWS could be from Pb-containing dust fall-out, which reaches the drainage system with rain. Concentration of Pb from such source would vary widely depending on traffic density, industrial emissions and climatic factors [42].

The Zn concentrations for the two wastewater treatment plants are below the limits provided by South Africa, USEPA and EU guidelines indicating again the relatively low risk the of the WWS samples with respect to Zn toxicity based on the regulatory limits specified in terms of the total Zn metal concentrations.

Zinc is an essential trace element for humans, animals and plants. However, high concentrations of Zn are potentially toxic to plants, humans and animals [43]. Though Zn has relatively low toxicity to humans and animals, studies have shown some allergies associated with Zn at high amounts and Zn poisoning could occur along the food chain, which may interfere with Cu metabolism [43]. Zinc can be derived from natural, domestic and industrial sources. Cities with metal industries and high traffic can contribute to high levels of Zn in sewage sludge [44].

Heavy metals have been proved to be toxic to environmental health. Wastewater sludge presented high quantities of nutrients, organic matters and almost neutral pH, appropriate to be used in agriculture. Heavy metals uptake by plants and successive accumulation in human tissues and biomagnifications through the food chain causes environment concerns.

Irrigation of agricultural soils by effluent and sewage sludge for several years increased HMs in soils and plants. The concentration of HMs increased significantly in the soil and plants of plots compared with control. Control of WWS pollution with HMs is therefore of great concern. Thus, it is essential to study the pollution sources of wastewater [45].

It has been mentioned by several researchers that limits based on total metals concentrations may be too restrictive as metals have different levels of mobility in soil and some of the HMs are present largely in the immobile fraction. Therefore, the regulatory limits based on total metals will have to be interpreted in terms of the relative mobility of the different heavy metals.

Further research is required with plant trials of the WWS applied in different soil environments and plants and with different degree of treatment of soils with WWS in order to determine the actual degree of toxicity of the HMs and their translocation within the plant structures. However, as it seemed from the result of the experiment in this investigation, that there is a good potential for the WWS from the wastewater treatment plants to be used for agricultural application. This positive information can be good news to the handling plants which presently store large loads of WWS in their WW treatment plants premises in globe with the possible future option of using the WWS for agricultural applications. Considering the nutrient and soil conditioning values of the WWS and reusing them for agriculture is an economical and environmentally sound option and deserves given greater attention.

Conclusion

This study investigated the effect of HM fractions in agricultural soils amended with WWS. The total metal concentrations present in WWS samples taken from the two wastewater treatment plants in Hungary showed a range of variations in accordance with the characteristics of the WWS generated from the respective cities and the level of industrial establishments present in the cities.

The WWS samples from Hódmezővásarhely wastewater treatment plant showed generally higher heavy metal concentrations compared with the two WWSs samples taken from the Nyíregyháza wastewater treatment plants. This is apparent as Hódmezővásarhely is an industrial area and several of the effluents generated from the industries have minimal treatments such as equalization basins before being discharged to the communal sewer system.

In terms of the regulatory limits of total metal concentrations mentioned in the USEPA, South Africa guidelines and EU directives, the sludge samples largely show compliance for agricultural application with respect to the majority of HMs. The experimental data by and large specified that most of the investigated HMs are present in moderate or at low concentrations well below the regulatory limits specified based on total metal concentrations and the WWS generated from the WW treatment plants may be considered further for agricultural application.

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