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Revegetation of Abandoned Sulphur Flotation Waste in Keçiborlu, Isparta/Turkey: Heavy Metals Concentrations of Growing Media and *Agropyron elongatum* Grass

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Abstract

The main objectives of the study are to evaluate the effectiveness of using amendment materials (calcareous soil and farmyard manure) and to grow *Agropyron elongatum* (tall wheat grass) for the revegetation of sulphur flotation waste in tailing ponds. Calcareous soil (CS) at 10, 20, 30, 40% and farmyard manure (FYM) at 4, 8% were applied to the flotation waste (FW). pH, electrical conductivity (EC), DTPA-extractable Fe, Zn, Mn, Cu, Ni, Cr, Pd, Cd, Co concentrations of growing media, also, total Fe, Zn, Mn, Cu, Ni, Cr, Pd, Cd, Co concentrations of plants were analysed together with the dry weight yield of the plant which were determined at end of the experiment. The results have shown that pH, shoot Zn, Mn, Cu and Pb concentrations increased by CS-alone but this increment tended to decrease by FYM. The EC, DTPA-extractable Fe, Zn, Mn, Ni, Cr, Co and shoot Fe, Ni, Cr, Co concentrations were decreased by CS-alone, but this decline tended to increase by FYM. There was no change in DTPA-extractable Pb and Cd concentrations between CS and CS with FYM. The dry weight of the plant shoots increased CS with FYM more than CS-alone. The plant grew well and had high cover in CS with FYM treatments compared to CS-alone. However this work indicates that *A. elongatum* can be used to revegetate sulphur flotation waste, further studies with different plant species are needed in order to obtain better plant cover in revegetation efforts.

Keywords: calcareous soil, farmyard manure, grass, inorganic waste, phytoremediation, reclamation, tailings

Introduction

Abandoned tailings which are the minerals processing residues around former mine plants constitute a major source of environmental contamination throughout the world. Tailings are often almost completely devoid of vegetation due to toxic levels of heavy metals and unfavourable edaphic conditions. The bare surfaces of tailings are susceptible to wind and water erosion and act as continuous source of metal contaminators to the surroundings (Shu *et al.*, 2005). The release of metals from mine sites takes place mainly through acid mine drainage and erosion of waste dumps and tailings deposits (Salomons, 1995). These pollutant effects can reach local and, in some cases, regional scales (Rybicka, 1996). In a study, investigating environmental effects of sulphur flotation waste ponds in Keçiborlu, Isparta/Turkey, an increasing concentration gradient for total and DTPA-extractable heavy metal in surface soil of both agricultural lands and centre of Keçiborlu district was detected toward the waste ponds (Yazar, 2010). Heavy metals in the soil of old mining areas, besides affecting the productivity of their ecosystems, could also affect animal and human health (Gutierrez-Gines *et al.*, 2010).

Many abandoned mines and their tailing ponds are barren or have minimal vegetation to slow natural colonization. Common physical and chemical limitations to plant growth on mine tailings include: low pH, high salt content, lack of organic matter and plant nutrients, metal toxicities, high bulk density, lack of soil structure, slow water infiltration, low water retention, and low air permeability (Henriquez and Fernandez, 1991; Wong *et al.*, 1998; Wong, 2003). Inorganic and organic amendments are useful for stabilization of mine soil prior to revegetation (Alvarenga *et al.*, 2008). Revegetation can be used for in situ reclamation of mine wastes, and this technique is also considered as the most suitable to achieve long term reclamation (Tordoff *et al.*, 2000). Vegetation can provide effective protection against wind carried polluted particles, reducing runoff and the overland flow of water and sediments. Vegetation may also improve nutrient conditions in the soil and form the basis for the establishment of a self-sustaining vegetative cover (Norland and Veith, 1995). Revegetation would require the addition of lime and/or organic matter to facilitate plant growth and increase the diversity of plant species (Conesa *et al.*, 2007).

Organic residues (e.g. manure, compost, sewage sludge) are increasingly being considered in land rehabilitation (Walker *et al*., 2004; Brown *et al*., 2005; Pérez-de-Mora *et al*., 2006). The addition of organic matter to mine soils can significantly improve the physical nature of the root medium, especially by improving water and nutrient holding capacity, supply of plant nutrients in a slow release form, facilitating plant establishment and in situ chemical immobilization of metals, reducing leachability and phytotoxicity (Tordoff *et al*., 2000).

Therefore, soil acidity is the limiting factor for the establishment of vegetation in mine wastes, it is common practice to apply liming materials to overcome some of the problems associated with soil acidity (Wong, 2003; Sutton and Dick, 1987; Ye *et al*., 1999). Alkaline amendments reduce the concentration of metals in soil solution by raising soil pH, thereby allowing the formation of insoluble metal precipitates, complexes and secondary minerals (Basta *et al*., 2001). Soils polluted with sulphur-mine wastes should be remediated by using inorganic materials containing $CaCO_3$ (higher than 40 g $CaCO_3$/kg dry soil) (Del Moral *et al*., 2010). The applications of calcareous soil and farmyard manure increased the soil's pH, decreased electrical conductivity (EC) and DTPA-extractable concentrations of Fe, Zn, Mn, Cu, Ni, Cr, Pb and Cd in sulphur mine tailings, and also increased shoot dry weight of *Zea mays* L. plant (Orman and Kaplan, 2007).

Choosing the plant material is very important for revegetation of mine wastelands. Plants should have to develop an extensive root system and a large amount of biomass in the presence of high concentrations of heavy metals, in order to keep translocation of metals from roots to shoots as low as possible (Wong, 2003; Rizzi *et al*., 2004; Mendez and Maier, 2008). Some *Agropyron* species, such as *A. desertorum* and *A. intermedium* have been used for mine reclamation (Williamson *et al*., 1982). *A. elongatum* has also been used to grow on salt-affected lands (Roundy, 1985).

The main objective of the present study was to evaluate the effects of a farmyard manure and calcareous soil in the remediation of a sulphur flotation waste as, also, called sulphur mine tailings and to determine the possibility of using *A. elongatum* (tall wheat grass) in its revegetation.

Materials and methods

Experimental materials

The abandoned sulphur flotation waste ponds of Keçiborlu Sulphur Factory were located in Keçiborlu/Isparta/Turkey (37° 57' N, 30° 18' E) with a mean annual rainfall 615 mm, which falls in winter and spring months. The wastes from the flotation of sulphur ores were stored permanently in the tailing ponds, approximately up to 1 million tons (Fig. 1). The sulphur flotation waste ponds requires revegetation to reduce the environmental impact, which is a major source of heavy metal pollution in the local environment, owing to dust below and from leaching of the products of mineral weathering into nearby watercourses. The sulphur flotation waste was collected from the tailing ponds form 0-20 cm depths. The extremely calcareous soil was taken from the campus area of Akdeniz University in Antalya, Turkey. The soils were classified as Lithic Xerorthent. The farmyard manure was supplied by a local farm. All samples (flotation waste, soil and farmyard manure) were air dried and screened through a 4 mm mesh sieve before use. *A. elongatum* (tall wheat grass) was grown as experimental plant.

Experimental design

The greenhouse pot trial was designed to test the growth of *A. elongatum* in sulphur flotation waste amended with calcareous soil and farmyard manure. A total of 6 kg of experimental materials (Flotation Waste (FW) + Calcareous Soil (CS) + Farmyard Manure (FYM) = Growing Media) at predetermined ratios (Tab. 1) were thoroughly mixed and then N:P:K fertilizer (15:15:15) was added at a rate equivalent to 150 kg da^{-1} per pot. The pots (510x165x125 mm) were filled up with growing mediums. There were prepared four replicates for each treatment.

Each pot was hand broadcasted with seeds *A. elongatum*, at rate of 100 g m^{-2}. The pots were arranged in a randomized factorial design and were equally watered in greenhouse and allowed to grow for 3 months (from the beginning of June - until the end of September). The above-ground parts (shoots) of the plants were harvested 5 times during the experiment. After harvest, plant samples were washed with tap water, rinsed three times with deionized water and then oven-dried at 65 °C to a constant weight. Dry weight yields were recorded. The dried plants resulted from the 5 harvests were homogeneously combined together to obtain a composite plant sample for each treatment. After this process, plant samples were reduced to powder by an electric mill and analysed to determine the concentrations of Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd, Co. The growing media samples were taken from each pot at the end of the experiment, air-dried, grounded to pass through a 2 mm sieve, and determined the pH, EC (Electrical Conductivity) values and concentrations of DTPA-extractable Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd and Co.

Tab. 1. Experimental design for pot trial

Treatments	A total of 6 kg per pot (w/w)
$FWCS_{10}$	90% FW + 10% CS
$FWCS_{20}$	80% FW + 20% CS
$FWCS_{30}$	70% FW + 30% CS
$FWCS_{40}$	60% FW + 40% CS
$FWCS_{10}FYM_4$	86% FW + 10% CS + 4% FYM
$FWCS_{10}FYM_8$	82% FW + 10% CS + 8% FYM
$FWCS_{20}FYM_4$	76% FW + 20 %CS + 4% FYM
$FWCS_{20}FYM_8$	72% FW + 20% CS + 8% FYM
$FWCS_{30}FYM_4$	66% FW + 30 %CS + 4% FYM
$FWCS_{30}FYM_8$	62% FW + 30% CS + 8% FYM

Also, air-dried flotation waste, calcareous soil and farmyard manure were grounded to pass through a 2 mm sieve and some physico-chemical parameters were analysed prior to the plant growth experiment (Tab. 2).

Analytical chemistry

The pH and EC values were measured in H_2O (1-2.5 soil: deionized water). The $CaCO_3$ content was determined by using a Scheibler calcimeter. The total N was done by using modified Kjeldahl procedure (Kacar, 1994). The available P was extracted by sodium bicarbonate (Olsen and Sommers, 1982), exchangeable K^+, Ca^{+2}, Mg^{+2}, Na^+ were extracted by ammonium acetate (Kacar, 1994), extractable Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd and Co were extracted by diethylenetriaminepentaacetic

Fig. 1. General views of the sulphur flotation waste (sulphur mine tailings) in Keçiborlu, Isparta, Turkey

acid-DTPA (Lindsay and Norvel, 1978) and then concentrations were determined by Induced-Coupled Plasma with Optical Emission Spectrophotometer (ICP-OES).

Plant samples (shoots) were digested with HNO_3 and $HClO_4$ (4:1) (Kacar and Inal, 2008). The concentrations of Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd and Co in the digests were determined by ICP-OES.

The data were analysed by standard ANOVA procedures and their significances were based on the $p<0.05$ level using the Duncan's Multiple Range Test.

Tab. 2. General properties of sulphur flotation waste, calcareous soil and farmyard manure

Parameters	Flotation Waste	Farmyard Manure	Calcareous Soil
pH	3.12	7.62	7.97
EC dS m^{-1}	9.99	4.73	0.16
$CaCO_3$ %	1.61	9.66	48.25
N %	0.038	2.13	0.035
P mg kg^{-1}	0.17	3876.16	0.49
K mg kg^{-1}	0.01	6767.84	23.87
Ca mg kg^{-1}	1442.00	36362.10	1442.00
Mg mg kg^{-1}	406.00	5116.83	130.00
Na mg kg^{-1}	4.92	0.15	15.01
Fe mg kg^{-1}	6250.00	3582.50	3.53
Zn mg kg^{-1}	8.17	152.58	1.18
Mn mg kg^{-1}	53.21	75.58	3.00
Cu mg kg^{-1}	10.30	2.56	0.59
Ni mg kg^{-1}	64.84	28.09	0.11
Cr mg kg^{-1}	14.86	57.56	0.02
Pb mg kg^{-1}	0.53	5.35	0.16
Cd mg kg^{-1}	0.13	0.56	0.01
Co mg kg^{-1}	5.82	0.27	0.04

Results and discussions

The pH, EC values and DTPA-extractable metal concentrations in growing media

The results of pH, EC and DTPA-extractable metal concentrations of growing media were given in Tab. 3 and Fig. 2. The pH of growing media increased by CS-alone, but this increment tended to decrease by FYM. Also, the EC was decreased by CS-alone, but this decline tended to increase by FYM. When contaminated soil is moist and exposed to air, oxidation and hydrolysis of sulphides (S^{2-}) in the waste leads to the formation of sulphuric acid, thus lowering pH and greatly increasing heavy metal solubility (Williamson and Johnson, 1981; Simón *et al.*, 1999; Clemente *et al.*, 2003). The application of both lime and organic matter to contaminated soil by mineral sulphides caused increasing pH and decreasing EC in soil (Clemente *et al.*, 2003).

The concentrations of DTPA-extractable Fe, Zn, Mn, Ni, Cr and Co concentrations were decreased by CS-alone, but this decline tended to increase by FYM. There was no change in Pb and Cd concentrations between CS and CS with FYM. The application of calcareous soil led to increased pH while consequently extractable metal concentrations were decreased in growing media. The pH effect was greater than that of metal complex formation by soluble organic matter (Walker *et al.*, 2004). Application of fresh manure can increase heavy metal solubility, due to the provision of soluble organic compounds which form complexes with the metals (Almås *et al.*, 1999; Shuman, 1999). However, the humic materials, which constitute a major part of the organic matter of compost, can reduce metal solubility by formation of stable metal chelates (Ross, 1994). Differences with respect to effects on metal availability between amendment materials can be related to differences not only in the organic matter, but also in the mineral fractions (pH, salt content) and cation exchange capacity, as well as changes in the redox conditions of the soil (Shuman, 1999; Ross, 1994; Walker *et al.*, 2003).

Metal concentrations and dry weight yield of Agropyron elongatum shoot

The concentrations of metal in plant shoots were given in Tab. 4 and Fig. 3. The shoot Fe, Ni, Cr and Co concentrations were decreased by CS-alone, but this decline tended to increase by FYM. The shoot Zn, Mn, Cu and Pb concentrations increased by CS-alone, but this increment tended to decrease by FYM. The dry weight yield of plant increased CS with FYM more than CS-alone (Fig. 3). The plant grew well and had higher cover in CS with FYM treatments compared to CS-alone (Fig. 4). Farmyard manure is commonly used as tailings amendment because the addition of organic matter can significantly improve the physical characteristics and the nutrient status of mine wastes (Bradshaw and Chadwick,

Tab. 3. The pH, EC values and DTPA-extractable metal concentrations in flotation waste amended with calcareous soil (CS) and farmyard manure (FYM) (as called growing media)[a]

Parameters	$FWCS_{10}$	$FWCS_{20}$	$FWCS_{30}$	$FWCS_{40}$	$FWCS_{10}$		$FWCS_{20}$		$FWCS_{30}$		Significance levels[b]
					FYM_4	FYM_8	FYM_4	FYM_8	FYM_4	FYM_8	
pH	3.53 hı	5.23 e	6.52 b	6.58 a	3.50 ı	3.57 h	4.22 g	4.40 f	5.43 d	6.30 c	***
EC (dS m^{-1})	2.83 b	2.57 d	2.39 f	2.46 e	2.70 c	2.56 d	2.20 g	3.14 a	2.70 c	3.13 a	***
Fe (mg kg^{-1})	282.73 d	221.10 f	100.82 ı	113.70 h	330.55 b	324.48 c	330.07 b	354.35 a	235.95 e	194.18 g	***
Zn (mg kg^{-1})	1.16 de	0.58 g	0.70 fg	0.89 ef	1.58 bc	3.46 a	1.01 e	1.78 b	1.36 cd	3.36 a	***
Mn (mg kg^{-1})	27.13 d	22.62 ef	13.65 g	16.00 g	25.42 de	37.92 b	34.63 bc	46.59 a	20.36 f	31.21 c	***
Cu (mg kg^{-1})	0.96 g	1.72 d	2.22 c	2.46 b	1.12 f	0.95 g	1.52 e	1.67 d	2.49 b	2.91 a	***
Ni (mg kg^{-1})	6.84 d	5.47 e	3.93 g	4.70 f	9.45 b	11.22 a	9.00 b	7.76 c	4.90 ef	8.10 c	***
Cr (mg kg^{-1})	0.03 c	0.02 c	0.02 c	0.02 c	0.12 a	0.10 b	0.02 c	0.02 c	0.02 c	0.02 c	***
Pb (mg kg^{-1})	0.04 ef	0.08 d	0.13 b	0.15 a	0.04 f	0.05 ef	0.06 e	0.09 d	0.11 c	0.15 a	***
Cd (mg kg^{-1})	0.03 e	0.05 c	0.05 c	0.06 b	0.03 d	0.04 d	0.04 c	0.05 c	0.05 b	0.08 a	***
Co (mg kg^{-1})	0.61 d	0.35 g	0.33 gh	0.46 ef	1.39 b	1.77 a	0.96 c	0.49 e	0.25 h	0.37 fg	***

[a]Values are means (n=4). Values in a row followed by different letters indicate significant differences (p<0.05) between treatments according to a Duncan's multiple range test.
[b]Significance levels: *** p< 0.001

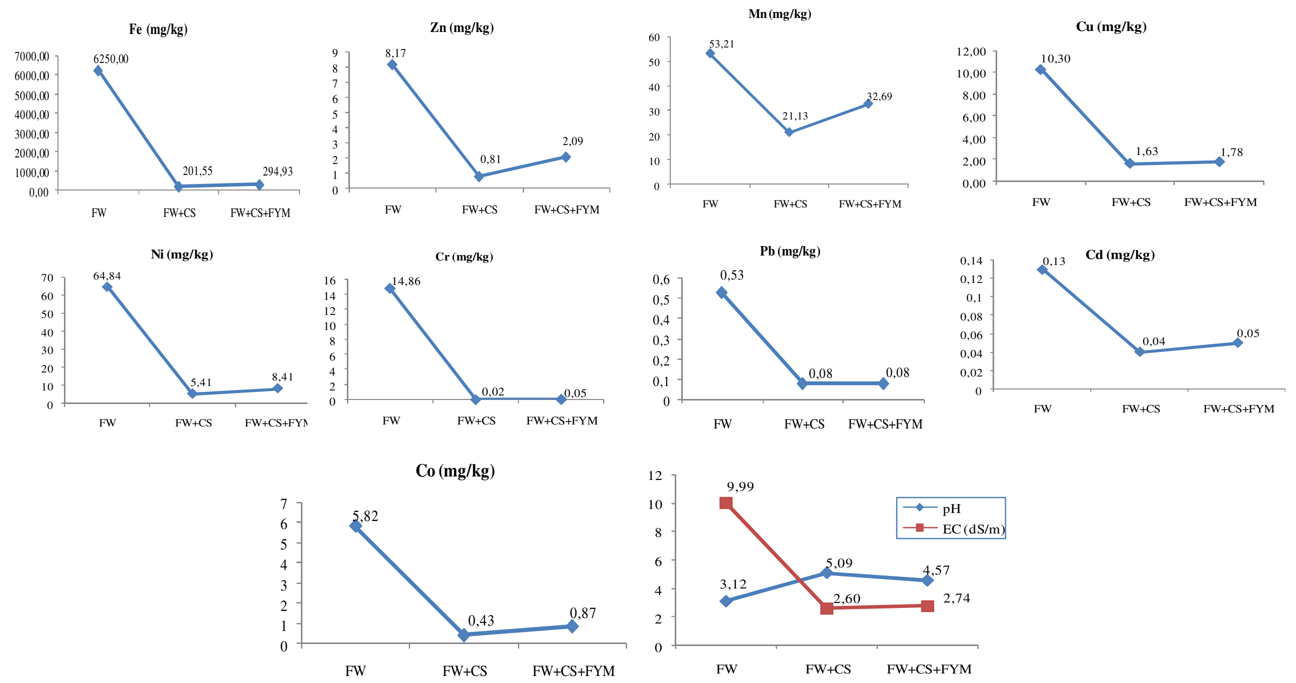

Fig. 2. The values of average pH, EC and DTPA-extract. Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd, Co concentrations due to treatments of calcareous soil and calcareous soil with farmyard manure in growing media

Tab. 4. Metal concentrations of *Agropyron elongatum* shoot growth in flotation waste amended with calcareous soil (CS) and farmyard manure (FYM) (as called growing media)[a]

Parameters	$FWCS_{10}$	$FWCS_{20}$	$FWCS_{30}$	$FWCS_{40}$	$FWCS_{10}$		$FWCS_{20}$		$FWCS_{30}$		Significance levels[b]
					FYM_4	FYM_8	FYM_4	FYM_8	FYM_4	FYM_8	
Dry weight (g pot^{-1})	25.34 h	29.61 g	35.33 e	32.57 f	31.85 f	40.25 d	40.31 d	46.94 b	41.79 c	48.54 a	***
Fe (mg kg^{-1})	275.80 d	256.13 e	239.32 gh	246.18 f	241.90 g	338.20 a	272.70 d	326.32 b	296.77 c	237.97 h	***
Zn (mg kg^{-1})	60.64 f	75.10 a	76.14 a	71.23 bc	66.89 d	70.06 c	69.27 cd	73.63 ab	62.52 ef	64.07 e	***
Mn (mg kg^{-1})	143.89 c	127.18 d	106.86 f	103.56 f	157.85 b	196.07 a	117.72 e	82.56 h	87.00 g	76.52 ı	***
Cu (mg kg^{-1})	1.56 c	1.60 b	1.35 fg	1.64 b	1.32 g	1.48 d	1.46 de	1.37 f	1.74 a	1.43 e	***
Ni (mg kg^{-1})	15.45 c	3.22 d	2.25 de	1.83 e	41.45 a	27.54 b	3.12 d	3.26 d	2.32 de	2.48 de	***
Cr (mg kg^{-1})	1.88 d	1.47 f	1.75 e	1.78 e	1.16 g	2.02 c	1.85 d	2.55 a	2.59 a	2.29 b	***
Pb (mg kg^{-1})	7.82 d	7.76 d	14.81 a	10.71 c	8.51 d	12.61 b	7.99 d	5.50 e	3.21 f	5.43 e	***
Cd (mg kg^{-1})	0.61 f	0.67 e	1.05 b	0.86 c	0.85 c	1.90 a	0.84 c	0.76 d	0.37 h	0.54 g	***
Co (mg kg^{-1})	1.16 c	0.32 de	0.35 d	0.31 e	3.86 a	1.50 b	< LD[c]	< LD	< LD	< LD	***

[a]Values are means (*n*=4). Values in a row followed by different letters indicate significant differences (p<0.05) between treatments according to a Duncan's multiple range test.
[b]Significance levels: *** p< 0.001
[c] Limit of detection (LD<0.1 mg kg^{-1})

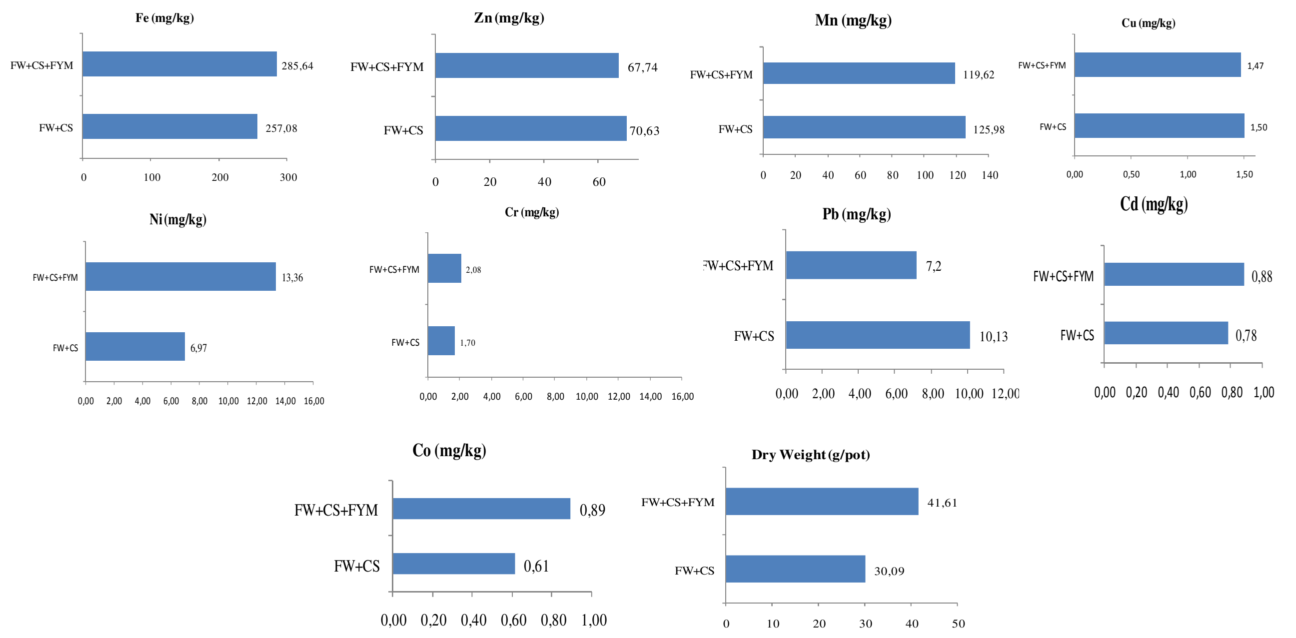

Fig. 3. The values of average Fe, Zn, Mn, Cu, Ni, Cr, Pb, Cd, Co concentrations and dry weight of shoot due to treatments of calcareous soil and calcareous soil with farmyard manure in growing media

Fig. 4. *Agropyron elongatum* growth during the experiment

1980). In addition, both manure compost and sewage sludge contained high amounts of macronutrients (N, P, and K) and organic carbon content, which can compensate for the nutrient deficiencies in Pb/Zn and Cu tailings (Chiu *et al*., 2006). The effect of the organic amendments on the bioavailability of metals was difficult to observe due to the great variability of total metal concentration and pH. In any case, pH seemed to be a controlling factor in metal availability, to a greater extent than the organic amendments. Liming succeeded in controlling the soil acidification in a soil affected by acid, highly toxic pyritic waste (Clemente *et al*., 2003).

Conclusion

DTPA-extractable heavy metal concentrations decreased with increasing pH in growing media caused by the applications of CS-alone to flotation waste. However, in applications of CS with FYM, heavy metal concentrations in both growing media and shoot increased, in contrast to increasing pH of the growing media. The higher DTPA-extractable heavy metal concentrations in CS with FYM treatments compared to CS-alone is thought to be the result of chelating effect of farmyard manure. In spite of high heavy metal concentration in growing media and shoot, dry weight yield increased in plant in CS with FYM treatments. This may be due to the improvements in some growing media properties, the presence of inorganic flotation waste, as a result of farmyard manure (organic).

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