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Impact of wastewater application on magnetic susceptibility in Terric Histosol soil

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A b s t r a c t. In this work, we attempted to analyse the changes in magnetic susceptibility in Terric Histosol soil irrigated with municipal wastewater in a period of four years. Effects of different plants (poplar and willow), wastewater doses, depths, as well as the concentration of the elements and the total carbon content were tested. The study showed that systematic wastewater irrigation diminished magnetic susceptibility values in the top layer of soil. However, statistical analysis revealed that both doses of wastewater and growing plants did not have a significant impact on the magnetic susceptibility of obtained results. Magnetic susceptibility decreased significantly with the depth, in accordance with higher total carbon and lower content of magnetic particles. High correlation coefficients were found between magnetic susceptibility and Zn, Al_2O_3, Fe_2O_3, MnO content, whereas no correlation was observed for Cr, as well as for Pb.

K e y w o r d s: wastewater irrigation, magnetic susceptibility, total carbon, heavy metals

INTRODUCTION

Presence of magnetic minerals in soils may be inherited from the parent rocks (lithogenic origin), be related to pedogenesis processes (pedogenic origin), or could result from anthropogenic activities (Baghdadi *et al.,* 2011; Boyko *et al*., 2004). Minerals such as magnetite and maghaemite are classified as ferromagnetics that strongly affect the value of magnetic susceptibility (Aleeksev *et al.,* 2002; Lu *et al.,* 2012b). Examples of paramagnetic minerals are biotite and pyrite. Their magnetic properties are caused by the presence of iron and manganese ions (Dearing, 1999). Goethite and hematite have antiferromagnetic properties (Strzyszcz *et al.,* 1994), while quartz, chalk, calcium carbonate, and orthoclase are diamagnetic minerals (Dankoub *et al.,* 2012). Non-mineral diamagnetic substances present in soil include organic matter, water, and plants with negative or weak magnetic susceptibility.

Anthropogenic contribution to magnetic susceptibility is a predominant factor in the organic layers of top soils (Schibler *et al.,* 2002). Up to now, magnetic susceptibility measurements have been widely used to evaluate the spatial distribution of pollutants (Lu *et al.,* 2008) in the upper layers of soil in industrial areas (Blundell *et al.,* 2009; Duan *et al.,* 2010), close to roads (Baghdadi *et al.,* 2011; Wawer *et al.,* 2015), and in marine and river sediments (Chan *et al.,* 1998; Chaparro *et al.,* 2011; Prajith *et al.,* 2015; Zan *et al.,* 2015). Several studies showed a strong correlation between magnetic parameters and the content of heavy metals (Karimi *et al.,* 2011; Ma *et al.,* 2015; Rosowiecka and Nawrocki, 2010; Strzyszcz and Magiera, 1998). However, it is still difficult to explain the mechanisms of these correlations in detail because they depend on a variety of complex and coactive physicochemical, pedological, or environmental factors (Lu *et al.,* 2012a; Zawadzki *et al.,* 2009).

Irrigation with municipal wastewater (WW) is considered to be an environmentally sound wastewater disposal practice compared to its direct disposal to the surface or ground water bodies. It can resolve certain environmental problems mainly related to an excessive or unbalanced supply of nutrients and the introduction of pollutants to ground water. In fact, not only biogenic substances retained in wastewater as residuals, but also a large amount of water can be recycled. On the other hand, continued irrigation may alter soil microbial activity and soil physicochemical characteristics, including changes in magnetic properties,

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organic matter, and heavy metal content. Accumulation of undesirable chemical constituents has potential to restrict some soil functions and cause plant toxicity and food chain contamination.

The purpose of this paper is to analyse changes in magnetic susceptibility in Terric Histosol soil irrigated with municipal wastewater for 4 years. Effects of different plant, wastewater doses, depths as well as concentrations of the elements and total carbon content are tested.

MATERIALS AND METHODS

The experiment sites were situated in the vicinity of a Wastewater Sewage Treatment plant in Lublin (South-East Poland). At this location, soil was classified as a peat-muck (Terric Histosol), according to the FAO classification. The basic soil properties were as follows: organic matter (OM) content 30.3-47.9%; pH 7.80-9.13 (in $CaCl_2$); bulk density 0.307-0.627 mg m^{-3} (Brzezińska *et al.,* 2011). The experimental area was divided into two plots of 1 hectare area. Each of them was planted with poplar (*Populus nigra* and *Populus alba* – plot 1) and willow (*Salix viminalis* and *Salix Americana* – plot 2) and then divided into three subplots (A, B, and C). The first subplot was marked as a control and received only natural precipitation. Subplots B and C were irrigated with municipal wastewater in two doses (60 and 120 mm). Irrigation was performed ten times in the vegetation period for four years. The value of the dose was established in accordance with the requirements of plants for nutrients (N, P, K) and water availability. The wastewaters were obtained after two-step mechanical and biological treatment. The main characteristics of wastewater (g m^{-3}) included: chemical oxygen demand (COD) 30.1-56.3; biological oxygen demand (BOD_5) 8.3-22.6; $N\text{-}NH_4^+$ 1.0-7.1; $N\text{-}NO_3^-$ 20.2-38.4; N_{tot} 22.3-43.6; $P\text{-}PO_4^{3-}$ 3.1-6.8; P_{tot} 3.7-7.0; Na^+ 24.3-69.4; K^+ 11.8-27.7; Ca^{2+} 59.7-95.2; Mg^{2+} 12.6-19.7; SO_4^{2-} 43.6-116.3; Cl^- 67.8-121.6; Zn 0.018-0.800; Cu 0.006-0.198; Pb 0.007-0.096 (Kotowski *et al.,* 1999), and pH in the range of 6.5-8.4. Soil samples were taken from the depths of 0-10, 10-30, 30-50, and 50-70 cm, three years after the period of wastewater application. Three places of sampling located on each subplot were chosen randomly. Before analysis visible fractions of roots and mesofauna were removed. The soil was air-dried and sieved through a 2 mm mesh.

The elemental analyses were performed using a desktop XRF crystal diffraction scanning spectrometer SPECTROSCAN MAKC-GV1 (Alekseeva *et al.,* 1999). The concentrations of Cr, Zn, Pb, MnO, Fe_2O_3, and Al_2O_3 were investigated. The quantitative analysis method was based on 12 standard soil calibration samples and then optimized using a computer simulation program.

The magnetic susceptibility (MS) was studied using the Kappabridge KLY-2 (Alekseev *et al.,* 2002).

Total carbon content (C_{tot}) was determined by combustion of samples under an oxygen atmosphere at 1250°C. A TOC MULTI N/C 2000, HT 1300 (Analytic Jena) device was used.

Statistical analysis was performed using Statistica 10.0 (StatSoft, Inc., Tulsa, USA). One-way analysis of variance (ANOVA) and post-hoc analysis (HSD Tukey test) were performed to test the influence of three factors (dose, depth, and plant) independently for the measured MS values. The F-value distribution was used to test the variance of magnetic susceptibility. Moreover Pearson correlation coefficients between means of MS values, heavy metal concentrations, and total carbon content were also determined. The significance level was evaluated at $p<0.05$.

RESULTS AND DISCUSSION

The concentration of heavy metals is presented in Table 1. Chemical analyses showed that the concentration of Zn decreased in the soil profile. The greatest quantity of the metal was noticed for the high doses of sewage – 75.5 and 71.5 mg kg^{-1}, respectively, for the plots planted with poplar and willow. In contrast to Zn, the concentrations of Cr and Pb were homogeneous with no effect of the doses and plants. The average values of the Cr content oscillated between 55.8 and 44.1 mg kg^{-1} and in the case of Pb from 108.3 to 59.3 mg kg^{-1}.

The content of metal oxides is presented in Table 2. The amount of Fe_2O_3 and Al_2O_3 decreased in the soil profile, irrespective of the wastewater dose. However, it slightly diminished for the plots with poplar. In the case of MnO, both doses of WW significantly affected the oxide content. The highest average value (1 503 mg kg^{-1}) was obtained in the control plot planted with willow at the depth of 0-10 cm. The lower dose caused a decrease by 51% whereas the higher dose – by about 47% within the same layer. The same effect was observed for the plots planted with poplar. The average value of MnO decreased by 58 and 38% for the low and high doses, respectively. At the depth of 30-70 cm, the concentration of MnO ranged from 631 to 397 mg kg^{-1} and was similar for all plants and doses. The decrease observed in the MnO content may be related to the fact that Mn is relatively mobile in organic soils, due to the weak bonding with soil OM (Kabata-Pendias and Pendias, 1999). Moreover, a small amount of clay minerals present in the investigated soil may cause low absorption of Mn into their crystal lattice or adsorption on exchange sites (Bradl, 2004). Therefore, higher elution of Mn in top layers might occur during wastewater irrigation.

Within the investigated area, the highest values of MS were observed for the top layer of soil and decreased values with increasing soil depth. This difference can be explained by the higher content of iron oxides in the top layer of the soil. A high concentration of ferrimagnetics was found in the top soil Of (fermentation) and Oh (humic) horizons (Zawadzki

T a b l e 1. Contents of heavy metals in Terric Histosol soil under poplar and willow

Depth (cm)	Cr			Zn (mg kg^{-1})			Pb		
	Control	Low	High	Control	Low	High	Control	Low	High
Soil planted with poplar									
10	55.8	48.7	55.0	62.9	52.1	75.0	74.5	66.9	87.3
	(5.3)	(4.5)	(5.0)	(4.2)	(3.6)	(4.0)	(16.4)	(14.6)	(15.6)
30	48.4	53.6	49.6	48.1	39.1	31.6	105.1	86.8	81.4
	(5.1)	(5.0)	(5.4)	(4.0)	(3.8)	(4.3)	(16.5)	(16.2)	(17.8)
50	53.1	54.1	43.1	27.7	26.0	33.9	100.7	108.3	75.9
	(5.1)	(5.5)	(5.4)	(3.9)	(4.4)	(4.4)	(16.7)	(18.4)	(18.1)
70	52.7	55.8	45.7	34.2	34.2	29.7	86.8	105.5	86.0
	(5.0)	(5.0)	(5.4)	(3.9)	(3.8)	(4.4)	(16.5)	(16.2)	(18.6)
Soil planted with willow									
10	47.6	52.4	53.9	67.5	50.6	71.5	77.4	92.2	59.3
	(5.0)	(4.8)	(5.9)	(4.1)	(3.7)	(4.7)	(15.9)	(14.6)	(18.3)
30	42.2	51.7	50.0	34.5	49.7	32.7	82.5	83.3	72.7
	(6.0)	(5.0)	(5.1)	(4.9)	(3.8)	(3.9)	(20.2)	(15.2)	(16.4)
50	41.7	55.4	47.7	35.0	42.3	25.0	78.0	105.9	89.5
	(5.3)	(5.4)	(4.9)	(4.4)	(4.2)	(3.9)	(18.0)	(17.4)	(16.3)
70	41.1	50.5	54.1	21.5	26.9	40.4	74.9	101.9	76.3
	(3.9)	(5.1)	(5.0)	(3.3)	(4.0)	(3.8)	(14.2)	(16.9)	(16.0)

Average values with standard error in parenthesis.

et al., 2010). On the other hand, organic matter possesses diamagnetic properties and affects weak and negative values of magnetic susceptibility (Dearing, 1999). The total carbon content slightly increased in the soil profile with no significant response to WW application with average values of 217 mg g^{-1}. It is worth mentioning that the average values of MS for the control variants in the top layer of the soil were 19.7 and 18.4 10^{-8}. Simultaneous wastewater application diminished the measured MS. In the case of the irrigated plots, the values oscillated between 5.6 and 11.7 10^{-8} (for poplar and willow, respectively). This effect was probably related to elution of some ferrimagnetic components together with the dissolved fraction of WW organic matter and with their movement through the soil profile (Lu *et al.,* 2012b). The statistical analysis performed (one way ANOVA, at a level of significance 0.05) showed that the differences between the wastewater variants and plants were not statistically significant. The F-values were 0.69 ($p=0.41$) and 2.51 ($p=0.09$) for the plant and dose variant, respectively. However, it should be noticed that the value of $p=0.09$ obtained for the effect of the dose is close to the given significance level. In this case, the relatively small number of samples used in statistical analysis may affect correct interpretation of the results. Therefore, for better understanding of the impact of wastewater dose variants on MS, as well as to avoid possible measurement errors, more repetitions are required. The depth had an influence on the magnetic parameter, especially in the layers of 0-10 and 10-30 cm (F-values equalled 37.55 at $p<0.05$). At the depth of 30-70 cm, no differences between the MS values were observed (at $p<0.05$). Deeper layers (30-70 cm) in the soil profile were represented through the lithogenic background of magnetic susceptibility and showed contribution to soil MS (Wang and Qin, 2005). At this depth, magnetic susceptibility is almost constant or varies slightly. In accordance with Hanesch and Scholger (2002), soil processes act within this layer while anthropogenic impacts are constrained to the upper horizons.

T a b l e 2. Contents of MnO, Fe_2O_3, and Al_2O_3 in Terric Histosol soil under poplar and willow

Depth (cm)	MnO			Fe_2O_3 (mg kg^{-1})			Al_2O_3		
	Control	Low	High	Control	Low	High	Control	Low	High
				Soil planted with poplar					
10	1 040.3	436.5	645.2	1.9	1.0	1.4	5.3	3.8	5.0
	(21.96)	(12.59)	(16.18)	(0.02)	(0.01)	(0.02)	(0.35)	(0.28)	(0.33)
30	925.9	463.2	520.2	1.4	0.7	0.8	4.6	3.2	3.4
	(20.82)	(13.89)	(15.81)	(0.02)	(0.01)	(0.01)	(0.34)	(0.29)	(0.31)
50	631.3	458.8	417.3	0.7	0.4	0.4	2.4	2.6	2.7
	(16.65)	(15.38)	(14.60)	(0.01)	(0.01)	(0.01)	(0.28)	(0.30)	(0.31)
70	488.9	504.0	397.4	0.5	0.6	0.5	2.8	2.6	3.0
	(14.52)	(14.53)	(14.15)	(0.01)	(0.01)	(0.01)	(0.30)	(0.28)	(0.31)
				Soil planted with willow					
10	1 502.9	731.7	791.9	2.1	2.2	1.8	4.8	6.5	5.9
	(27.60)	(16.89)	(20.29)	(0.02)	(0.02)	(0.02)	(0.33)	(0.34)	(0.38)
30	1111.1	681.9	530.6	0.9	2.1	0.9	3.0	6.7	3.6
	(26.29)	(16.80)	(15.14)	(0.01)	(0.02)	(0.01)	(0.35)	(0.36)	(0.31)
50	550.8	560.7	488.5	0.6	0.7	0.8	2.5	2.9	3.0
	(16.40)	(16.21)	(14.56)	(0.01)	(0.01)	(0.01)	(0.30)	(0.32)	(0.29)
70	568.5	426.5	435.1	0.9	0.5	0.9	2.5	2.3	3.7
	(12.85)	(13.98)	(13.40)	(0.01)	(0.01)	(0.01)	(0.20)	(0.28)	(0.30)

Average values with standard error in parenthesis.

The plots presented in Fig. 1 show correlations between magnetic susceptibility and the content of total carbon, heavy metals, and oxides of Mn, Fe, and Al, as well as the regression equations for each pair. The Pearson correlation coefficients were calculated for all samples as a single group including all depths.

The total carbon content showed a negative relation with MS ($R=-0.46$, $p<0,05$) and this could reflect the distribution of organic matter in the soil profile. Organic matter is mentioned among the natural factors that can induce a decrease in magnetic susceptibility (Thompson and Oldfield, 1986).

Linear and positive correlations were confirmed for Zn ($R=0.75$, $p<0.05$), MnO, Fe_2O_3, and Al_2O_3 ($R\geq0.76$, $p<0.05$). The high correlation is associated with the presence of feromagnetic components in the soil, especially the crystalline iron oxides, for which the greatest correlation was observed ($R=0.89$, $p<0.05$). These components are common in the soil, but they can have an anthropogenic origin.

In accordance with previous study (Duan *et al.,* 2010), the high correlation between MS and heavy metals like Zn, Pb, and Cu can be attributed to the absorption into ferrimagnetic minerals, already present in the environment. In the solute form, these metals show similar chemical properties and have a tendency to co-precipitate with hydrous oxides of Fe and Mn (Chan *et al.,* 1998; Wang and Qin, 2005). On the other hand, no correlations were observed between MS and Cr ($R=0.17$, $p<0.05$) and Pb ($R=-0.20$, $p<0.05$). Cr did not show an anthropogenic increase. This element should be treated as a common factor with distributions in the soil being lithologically controlled (Dankoub *et al.,* 2012; Huliselan *et al.,* 2010). No correlation between Pb and MS was observed by other authors (Gelisli and Aydin, 1998; Schmidt *et al.,* 2005). However, the cause of such weak dependence could be the small number of replicates as well as the combination of samples from four analysed depths.

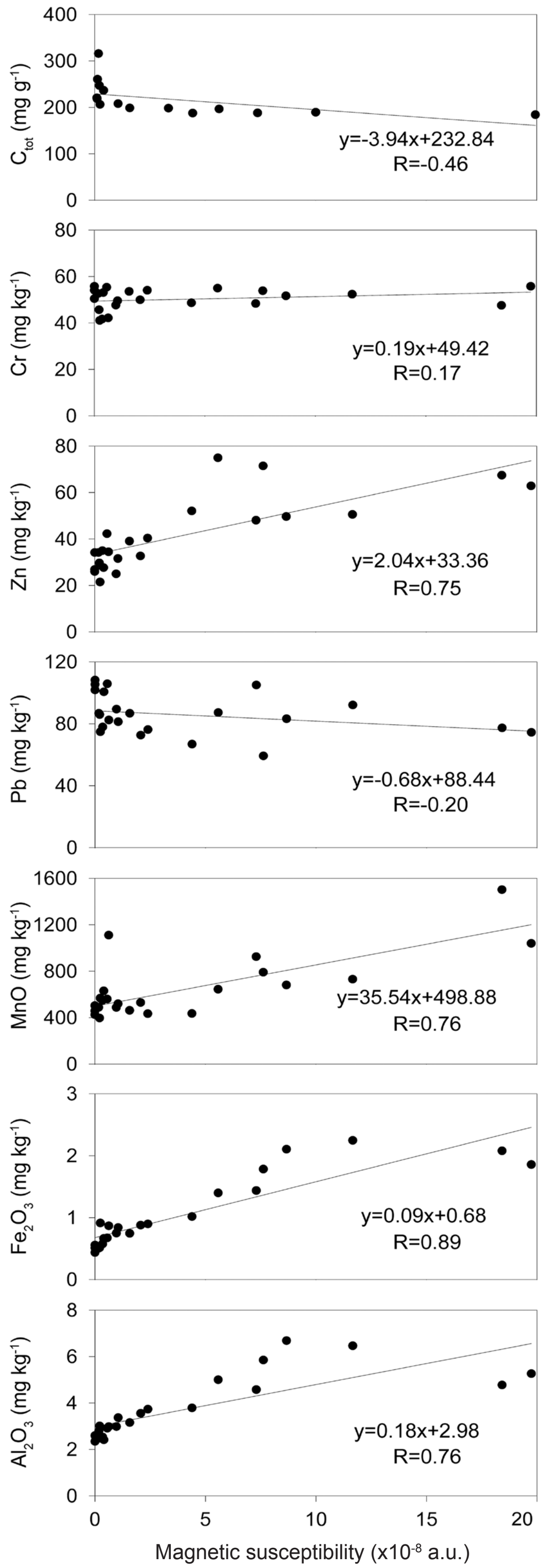

Fig. 1. Scatter plots and regression equations between magnetic susceptibility and C_{tot}, Cr, Zn, Pb, MnO, Fe_2O_3, Al_2O_3 content. Pearson correlation coefficients for each pairs were calculated for all samples collected in the studied area.

CONCLUSIONS

Magnetic susceptibility measurements are both fast and convenient and can be used to evaluate and monitor potential soil changes during wastewater irrigation. The main conclusions from this study are as follows:

1. Simultaneous wastewater application diminished the measured magnetic susceptibility Terric Histosol. However, statistical analysis showed that the differences between the wastewater variants and plants were not significant at p=0.05. The depth had also an influence on the magnetic parameter. The highest values of magnetic susceptibility were observed for the top layer of soil; they decreased when the soil depth increased.

2. Linear and positive correlations were confirmed for Zn (R = 0.75, $p < 0.05$) and all the studied oxides ($R \geq 0.76$, $p < 0.05$) with a negative relation for C_{tot} (R=-0.46, $p < 0.05$). No correlation between magnetic susceptibility and Cr and Pb was observed. The number of replicates as well as the combination of samples from all depths could affect the Pearson correlations coefficient obtained.

3. The manganese oxide content decreased significantly after wastewater application. This is connected with the high mobility of MnO, its weak binding with soil organic matter, as well as the small amount of clay minerals in the investigated soil.

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