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ORIGINAL PAPER

THE IMPACT OF LAND USE ON THE ORGANIC AND INORGANIC SELENIUM CONTENT IN SOILS DEVELOPED FROM LOESS*

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Abstract

The mobilisation of selenium into the biosphere by human activity has become an important process in the geochemical cycling of this element. This study reports the Se content in elemental, exchangeable and organic-bound forms by conducting sequential extraction of soil and evaluation of selenium behaviour in soils developed from loess. The evaluation of Se immobilisation and mobilisation factors in soils included the role of soil in areas with diverse land use and high anthropogenic pressure. Lublin, where the study was conducted, was divided into 5 areas according to the land use pattern. i.e.: industrial areas (IA), areas used by transport (TA), housing estates (HA), municipal gardens (GA), farmland (FA), municipal forest (FC) and two benchmark profiles: arable farmland area (FAR), and forest area (FCR) in Czesławice n. Nałęczów, that is outside of the Lublin agglomeration impact zone. The results of Se fractionation in soil indicated that industrial pollution (IA) and heavy fertilisation and pesticide use (GA) exert an impact on the mobilisation and transformation of most of this element in the environment. The mean Se total content in urban soil is in the range 0.81 - 0.397 mg kg^{-1}. The distribution of Se concentrations in soil profiles demonstrated a tendency towards Se attaining the highest values in the topsoil, while decreasing with depth. Exchangeable and oxidised organic-bound forms correlate with the organic and mineral content of 1-0.1 mm fraction. No correlation between the selected Se forms and the soil's silt fraction was observed; in contrast, a negative correlation was noted with the fine clay fraction.

Keywords: silt soils, speciation, micronutrient, anthropopressure, urban soils.

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INTRODUCTION

Despite its toxicity, selenium is a chemical element of great importance to humans, animals and certain microorganism species. It is included in over 30 proteins, called selenoproteins, which increase resistance of cells to free radicals, play a role in thyroid hormone metabolism, are involved in human and animal reproduction as well as inflammatory responses, and stimulate the nervous and immunological systems. An excess of selenium, however, is toxic to humans and results in chronic alkalosis or acute blind staggers (Brodowska et al. 2016).

The selenium range between deficiency and excess is fairly narrow, hence, taking into consideration disproportions in the selenium distribution in soils around the world, numerous researchers have explored the effects of selenium deficiency and toxicity in humans and animals (Brodowska et al. 2016). Through the food chain, the Se concentration in soil translates to its content in food. Reactivity and bioavailability of selenium in soil depend are not only on its total content but also on the chemical forms in which it is present in soil (Wells 1967). Depending on the degree of oxidation, selenium in soil takes the following forms: inorganic selenides, elemental selenium, selenites, selenates (Geering et al. 1968); there are also organic-selenic combinations in the soil. Selenium in all its forms is easily transformed under the influence of changing soil community conditions (Fernández-Martínez, Charlet 2009, Tolu et al. 2014).

The bioassimilability and migration velocity in soil profiles are important marks of the Se presence in soil. This study was based on speciation analysis, which enables determination of different selenium forms in soil (e.g. exchangeable, organic-bound, bound to crystal structures of soil minerals, *etc*) and is highly important from the ecological perspective in the evaluation of Se deficiency or contamination risks (Bujdoš et al. 2005). In general, only a certain percentage of total selenium content in soil is assimilated by plants or migrates to groundwater. It is estimated that 15-40% of selenium remains in the strongly-bound form, whereas water-soluble Se, bioavailable for plants, constitutes 6-10%. Sequential extraction is a frequently applied method of Se fraction determination in soil (Kang et al. 1993, Tolu et al. 2011, Wang et al. 2012).

Urban areas are characterised by a high degree of anthropogenic activity, such as intensive land use, often with numerous emission sources (Makuch 2014, Hulisz et al. 2017). The previous research on the behaviour of different pollutions in urban soils and their bioavailability is supplemented with investigations which additionally take into consideration the types of land use (Mendyk, Charzyński 2016). Anthropogenic sources of Se include industrial emission (predominantly through coal combustion), phosphorous fertilisers and municipal waste. The Se concentration in highly contaminated areas in Poland has risen by 0.060-0.818 mg kg^{-1} (Biernacka, Małuszyński 2006).

Research on selenium in different parts of the world either focuses on determination of selenium levels in total native soil content or different forms of this element depending on local emission sources, with reference to different limits and norms regulating risk levels for human health (Rodriguez et al. 2005). Research into the Se concentration in urban soils accounting for land use and function of urban soils in urban ecosystems will constitute significant expansion of former studies on the behaviour of Se in soil and health risk evaluation (Shang et al. 2015).

The present study was an attempt to determine the Se content in elemental, exchangeable and organic-bound forms by conducting sequential extraction of soil and evaluation of selenium behaviour in soils developed from loess. The evaluation of Se immobilisation and mobilisation factors in soils included the function of soil in areas with diverse land use and high anthropogenic pressure. This paper analyses lessive soils of different function and use, such as:

– forest within the city's area,
– arable lands,
– industrial area soils,
– fallow land in the vicinity of a road with high traffic density,
– agricultural wasteland transformed into a housing estate,
– soils under allotment gardens,
– benchmark forest profile,
– benchmark arable land profile.

MATERIAL AND METHODS

The survey was carried out in Lublin, which is a city located in the eastern part of Poland (N 51°14′; E 22°33′), in the Lublin Upland on the Bystrzyca River. The Bystrzyca valley divides Lublin into two parts: (i) the left-bank area with loess cover, ravines and dry valleys, and the right-bank area (ii), lower and gentler, built from Upper Cretaceous rock formations (marls, opoka, gaizes) and Palaeocene formations (gaizes) with Pleistocene sandy silts and loess-like silty sands.

Soils found in the Lublin region are mineral soils developed from loess, which are low in the Se content, which ranges between 0.17 and 0.34 mg kg^{-1}. Se deficiency in arable fields is often supplemented by fertilisation (Plak et al. 2004).

The soil samples were collected in different parts of the city. Although functionally dissimilar, they constitute lessive soils, developed from loess or loess-like deposits. The city was divided into 6 areas according to the land use pattern, i.e.: industrial areas (IA), areas used by transport (TA), housing estate (HA), municipal gardens (GA), farmland (FA), municipal forest (FC) and two benchmark profiles: arable farmland (FAR), and forest (FCR) in Czesławice n. Nałęczów, that is outside of the Lublin agglomeration impact zone.

In total, 49 soil samples representing particular genetic levels of the analysed profiles were collected and their physicochemical properties were determined. The fine clay fraction (<0.002 mm) was identified according to the Bouyoucos-Casagrande's aerometric method, modified by Prószyński, pH was determined in distilled water and 0.1 M KCl – potentiometrically, the $CaCO_3$ content was measured with the Scheibler`s volumetric metod (Ostrowska et al. 1991), while that of organic carbon was assayed using wet oxidation (Nelson, Sommers 1996). The sum of exchangeable bases – TEB (Ca, Mg, K and Na) was identified after extraction of the elements from soil with 1M ammonium chloride and applying the AAS technique. The Kappen method was applied to determine hdrolytic acidity (Hh). Cation exchange capacity (CEC) of soils is calculated as a sum of hydrolytic acidity (Hh) and total exchangeable bases (Ca, Mg, K, Na) – TEB (Ostrowska et al. 1991).

Sequential extraction of Se in soil was carried out by fractionation of its 3 forms: exchangeable – bioavailable for plants, organic-bound and residual (Bujdoš et al. 2005). The procedure is presented in Table 1.

Table 1

Procedure of the extraction of selenium forms from soils

Extraction conditions	**Extracted forms**
0.1 M (pH 7) K_2HPO_4-KH_2PO_4 (P-buffer), horizontal shaking for 1 h, centrifuging	soluble and exchangeable forms Se^{+6}, Se^{+4}, Se^{-2}
0.1 M $K_2S_2O_8$, heating for 2 h at 363 K	bound to organic matter Se^{-2}, Se^{+4}
Microwave mineralisation $HCl:HNO_3$; 3:1	residue associated with crystallic net of soil minerals

Mineralisation of the soil samples was conducted with *aqua regia* /nitro-hydrochloric acid/ using the ISO 11466 method. All selenium forms in soils were determined with sequential extraction and hydride generation method (HGAAS) on an AAS 3300 Perkin Elmer spectrometer equipped with a FIAS 400 flow injection system (Bujdoš 2000, Shand et al. 2010).

RESULTS AND DISCUSSION

Both urban silty soil profiles and benchmark profiles had developed from loess or loess-like deposits. The lessivage process is of importance as it allowed the formation of an illuvial Bt horizon, in which fine clay (<0.002 mm) is cumulated (Bt) – Table 2. The use and function of lands in a city determi-

Table 2

Basic properties of the analysed soil

Prof. No.	Horizon	Depth (cm)	Texture	C org. (g kg^{-1})	pH 1M KCl	Hh	TEB	CEC	BS (%)
						(mmol(+) kg^{-1})			
1	O	1-0	-	69.41*	4.98	467.7	277.8	745.5	37.26
	A	0-7	SiL	3.25	3.41	157.2	8.6	165.8	5.17
	AEet	8-15	SiL	0.79	3.75	378.8	3.4	382.2	0.89
	Bt	16-50	SiL	0.18	3.66	58.4	40.4	98.7	40.88
	BtC	50-70	SiL	0.14	3.83	32.9	42.5	75.4	56.36
	C	70-129	SiL	0.08	4.12	17.5	43.3	60.8	71.25
	Cca	<129	SiL	0.06	7.85	3.8	624.4	628.2	99.4
2	ApBt	0-15	SiL	0.78	4.66	60.5	51.9	112.5	46.16
	B1t	15-40	SiL	0.13	4.59	52.8	39.5	92.2	42.77
	B2t	40-65	SiCL	0.08	4.43	48.6	33.5	82.0	40.77
	BtC	65-90	SiL	0.04	4.42	18.5	50.8	69.3	73.35
	C	90-150	SiL	0.04	4.53	14.4	72.6	87.0	83.47
	Cca	<150	SiL	0.02	7.64	3.7	506.1	509.8	99.28
3	O	3-0	-	24.55*	4.92	196.5	175.4	371.9	47.17
	A	0-23	SiL	0.88	3.73	61.6	12.4	74.0	16.78
	Eet	23-48	SiL	0.4	3.82	44.4	5.8	50.2	11.51
	EetBt	48-62	SiL	0.11	3.87	35	20.7	55.7	37.19
	Bt	62-93	SiL	0.09	3.87	38	72.2	110.2	65.51
	BtC	93-160	SiL	0.06	3.97	27.2	73.0	100.3	72.85
	C	>160	SiL	0.05	3.96	21.6	64.3	85.9	74.88
4	Ap	0-33	SiL	0.77	3.88	50.5	50.1	100.6	49.77
	Bt	33-66	SiL	0.16	3.95	39.8	81.0	120.8	67.08
	Bt/C	66-82	SiL	0.08	4.05	19.5	49.5	69.0	71.76
	C	82-146	SiL	0.06	4.09	17.1	64.0	81.1	78.91
	Cca	<146	SiL	0.07	7.25	2.4	494.4	496.8	99.51
5	Ap	0-20	SiL	0.71	6.65	7.7	70.3	78.0	90.14
	Ap2	20-30	SiL	0.63	6.82	7.7	75.9	83.5	90.83
	IIAEet	30-36	SiL	0.18	6.88	4	65.9	69.9	94.24
	IIIBt	36-66	L	0.11	6.78	5.5	249.6	255.1	97.84
	IIIBt/C	66-71	SCL	0.17	7.01	5.8	465.0	470.8	98.77
	IIICca	> 71	L	0.19	7.32	5.2	478.9	484.2	98.92
6	Ap	0-20	SiL	1.04	7.07	6	153.4	159.4	96.24
	Bt1	20-50	SiL	0.22	7.45	2.4	208.1	210.5	98.84
	Bt2	50-70	SiL	0.16	7.44	2	115.7	117.7	98.28
	BC	80-100	SiL	0.09	7.23	1.6	118.0	119.6	98.65
	C	100-110	SiL	0.09	7.07	1.2	124.2	125.4	99.04
	Cca	>110	SiL	0.09	7.41	1.2	377.4	378.7	99.68
7	A	0-27	SiL	1.42	4.27	62.5	52.0	114.5	45.41
	Eet	27-37	SiL	0.89	4.95	38.5	81.0	119.5	67.78
	Bt	37-62	SiL	0.78	5.40	58.4	49.5	107.9	45.87
	Bt/C	62-75	SiL	0.32	5.23	18.5	50.8	69.3	73.35
	C	>74	SiL	0.12	5.38	14.4	72.6	87.0	83.47
8	A	0-30	SiL	1.72	4.30	39.8	66.1	105.9	62.41
	Eet/B	30 - 45	SiL	0.61	4.36	20.9	32.1	53.0	60.56
	Bt1	45- 60	SiL	0.43	4.20	26.0	51.6	77.6	66.49
	Bt2	60 - 80	SiL	0.32	4.16	27.6	77.9	105.5	73.84
	Bt/C	80 - 100	SiL	0.19	4.20	25.2	80.7	105.9	76.21
	C1	100 - 130	SiL	0.11	4.30	15.8	51.2	67.0	76.44
	C2	<130	SiL	0.07	4.28	15.4	59.2	74.6	79.35

nes the soil profile pattern. Forest soils profiles (FC, FCR) contain organic horizons O as well as eluvial E and AE horizons. In the remaining areas, where land is used differently, the eluvial horizon had been destroyed due to deep ploughing or soil transportation connected with works conducted in an urban area. Basic soil parameters are presented in Table 2. The soils are of silt-size grain.

The soil reaction showed significant differences and pH ranged between 3.41 and 7.85. Both benchmark profiles (FAR, FCR) and farmland area profile (FA) contained carbonates in the parent material. Carbonates violently effervescent with 10% HCl were found in subsurface horizons of the lessive soils in the industrial area (IA) and in the areas used by transportation (TA). The analysed soils showed significant differences in sorption capacity (CEC), which ranged between 50.2 and 745.5 mmol(+) kg^{-1}. Soil saturation with base cations was within a wide range of 0.89% in acidic forest soils to 99.68% in the parent material, which contained violently effervescent carbonates.

Essential statistical data on the Se content are collated in Table 3. Mean Se values in surface horizons were contained within the reference values for

Table 3

Basic statistics of selenium forms in particular functional areas (mg kg^{-1})

Statistics	**Se-ex**	**Se-org**	**Se-res**	**Se-tot**	Statistics	**Se-ex**	**Se-org**	**Se-res**	**Se-tot**
	reper/forest areas (FCR)					reper/field areas (FAR)			
Maximum	0.027	0.064	0.110	0.197	maximum	0.014	0.038	0.078	0.118
Minimum	0.002	0.014	0.055	0.072	minimum	0.001	0.006	0.029	0.036
Mean	0.008	0.029	0.085	0.121	mean	0.005	0.021	0.055	0.081
SD	0.009	0.018	0.022	0.046	SD	0.005	0.013	0.020	0.035
forest areas (FC)					field areas (FA)				
Maximum	0.024	0.056	0.139	0.178	maximum	0.018	0.047	0.113	0.167
Minimum	0.004	0.002	0.049	0.072	minimum	0.003	0.013	0.053	0.069
Mean	0.010	0.021	0.096	0.128	mean	0.008	0.029	0.083	0.120
SD	0.007	0.020	0.029	0.036	SD	0.006	0.014	0.025	0.037
industrial areas (IA)					traffic areas (TA)				
Maximum	0.100	0.234	0.314	0.648	maximum	0.036	0.088	0.125	0.249
Minimum	0.007	0.042	0.170	0.219	minimum	0.001	0.003	0.013	0.017
Mean	0.040	0.115	0.242	0.397	mean	0.010	0.031	0.069	0.109
SD	0.035	0.074	0.060	0.165	SD	0.013	0.032	0.050	0.091
garden areas (GA)					housing areas (HA)				
Maximum	0.081	0.175	0.279	0.516	maximum	0.028	0.076	0.136	0.225
Minimum	0.009	0.013	0.097	0.120	minimum	0.004	0.014	0.087	0.106
Mean	0.035	0.082	0.202	0.319	mean	0.010	0.030	0.104	0.144
SD	0.028	0.061	0.073	0.153	SD	0.008	0.021	0.019	0.043

silty soils of loess origin provided in Poland (Patorczyk-Pytlik, Kulczycki, 2009). In comparison, benchmark profiles collected outside the Lublin city influence zone were distinctly richer in Se than the soils found in the city area. Cisielczuk et al. (2011) investigated arable soils, neighbouring an industrial plant in Opole (Southern Poland), and noted the total content of selenium at a slightly higher level than in the industrial zone in Lublin, reaching 0.96 mg kg^{-1}. The selenium content in arable soils located in the vicinity of Wrocław, South-West Poland, amounted to 0.2 mg kg^{-1} on average, which was a similar level as in Lublin. The recorded values are lower than the mean concentration for Polish soils (Patorczyk-Pytlik, Kulczycki 2009). Urban settlements are areas of considerable anthropogenic activity, where numerous emission sources are present (e.g. transportation, coal combustion, industry), and simultaneously the soil use is intensive (Biernacka, Małuszyński 2006).

The soil profile in the industry area (IA) manifested a higher elemental Se content in comparison with the other soil types (Figure 1). A similar tendency was observed in the allotment garden area (GA). The lowest Se levels were noted in the areas used by transport, which is a direct indication of the fact that car pollution does not accumulate this element. The pattern of the mean Se content in Lublin's soils, of different use and function, follows the sequence: IA>GA>HA>FC>FA>TA. With regard to the profile distribution of elemental selenium in silt soils, the highest values are found in topsoils, with accumulation in the Bt horizon (Figure 1), where fine clays are found. High Se affinity to soil mineral fractions has been noted by numerous researchers (Hamdy, Gissel-Nielsen 1970, Wan, Chen 2003). However, statistical analysis showed no significant correlation between the elemental Se content and the colloidal fraction of the analysed soils (Table 3).

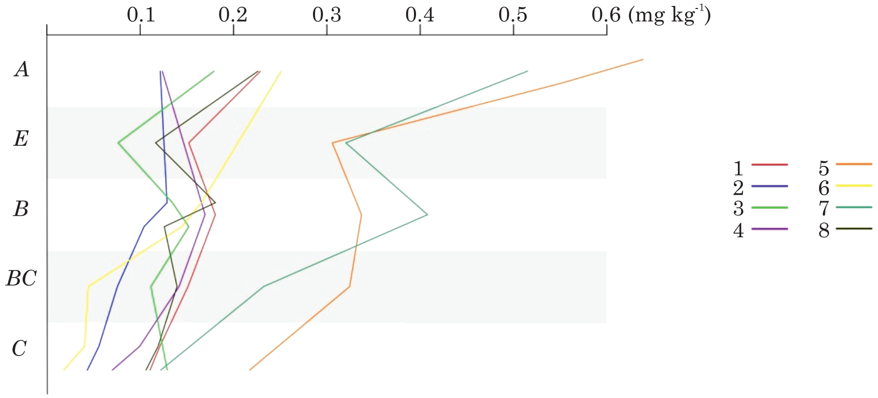

Fig. 1. Content of total selenium in analysed soils in terms of genetic horizons: 1-8 profile numbers

Speciation analysis of Se presence in silty soils of the Lublin area assists in an evaluation of the potential contamination of the local environment (Figure 2). The results of Se fractionation in soil indicated that industrial

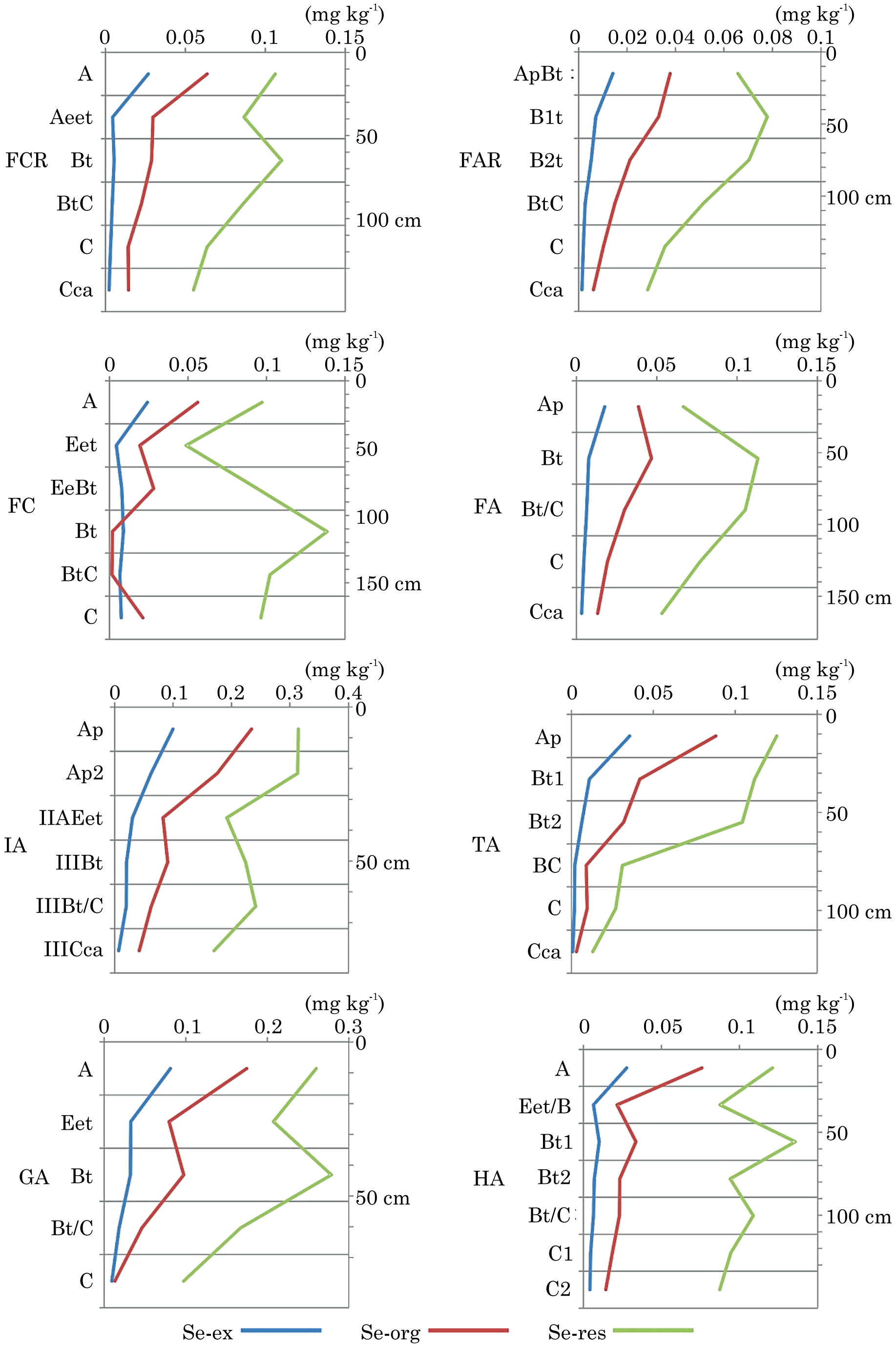

Fig. 2. Content of selenium forms in particular soil profiles in terms of genetic horizons: Se-ex – exchangeable Se, Se-org – organic Se, Se-res – residual Se

Table 4

Correlations between the content of particular selenium forms and chosen soil parameters

Parameter	Se exchangeable	Se organic	Se residual	Se total
1-0.1 mm	**0.47**	**0.57**	**0.67**	**0.63**
0.1-0.05 mm	0.01	-0.03	0.00	-0.01
0.05-0.02 mm	**-0.30**	**-0.41**	**-0.54**	**-0.48**
0.02-0.005 mm	0.04	-0.05	**-0.30**	-0.18
0.005-0.002 mm	0.28	0.25	0.15	0.21
<0.002 mm	**-0.42**	**-0.34**	-0.14	-0.26
Organic carbon	**0.48**	**0.44**	0.26	**0.37**
Hydrolytic acidity	0.00	0.01	-0.04	-0.02
Cation exchange capacity	-0.16	-0.12	-0.07	-0.10
Total exchangeable bases	-0.17	-0.12	-0.08	-0.11
Base saturation	-0.02	0.01	0.08	0.04

Marked values are significant at $p < 0.05$.

pollution (IA) and intensive fertilisation and pesticide use (GA) exert an impact on mobilisation and transformation of the biggest amounts of this element in the environment (SHANG at al. 2015) – Table 4. The first stage consisted of an extraction with phosphate buffer, which is efficient in removing soluble, absorbed and exchangeable Se compounds as well as selenium bound to organic protein. At the same time, the extractant removes the majority of humic and fulvic acids, which are partly soluble at pH 7. The observed correlation of this Se form with the organic content may indicate that in general organic compounds react with Se compounds. In the analysed soils, the exchangeable fraction was readily converted to immobilised forms, possibly due to the reduction to inorganic Se compounds and its elemental form.

Statistical analysis showed a negative correlation between exchangeable Se and organic Se with fine clay fraction (<0.002 mm). A distinct correlation, however, was observed between Se forms and the sand fraction (1-0.1 mm) – Table 4.

To a considerable extent, Se sorption depends on the soil reaction, which makes the surface of soil minerals constituting the 1-0.1 mm fraction and humic compounds to promote selenium sorption (SU, SUAREZ 2010). The Se speciation in soil has allowed researchers to obtain data on geochemical processes, probable reaction mechanisms of this element and the impact of such factors as pH or degree of oxidation on the chemical forms and mobility of Se in the soil environment (JOHNSSON 1991, HAGAROVA et al. 2005). The exchangeable selenium content depends on geochemical conditions and indicates an anthropogenic source of this element, either in the form of fertiliser or pollution (WENG et al. 2011, BRODOWSKA et al. 2016). The exchangeable form in the conducted study comprised 3.06% to 15.7% of the total Se content, and was found in the industrial area (IA) and allotment garden area (GA) soil profile (Table 5). Its concentration distribution in soil profiles demonstrated a tendency towards the highest values in the topsoil, decreasing with depth.

Table 5

Percentage of selenium in particular functional areas (mg kg^{-1})

Statistics	♀ **Se-ex (%)**	♀ **Se-org (%)**	♀ **Se-res (%)**	Statistics	♀ **Se-ex (%)**	♀ **Se-org (%)**	♀ **Se-res (%)**
	reper/forest areas (FCR)				reper/field areas (FAR)		
Maximum	13.6	35.3	78.9	maximum	12.0	32.1	79.3
Minimum	3.1	17.4	54.0	minimum	3.7	16.9	55.9
Mean	6.0	24.3	69.7	mean	5.8	23.7	70.5
forest areas (FC)				field areas (FA)			
Maximum	13.6	40.1	92.7	maximum	14.3	31.5	76.6
Minimum	5.9	1.2	50.2	minimum	4.5	18.9	54.2
Mean	7.6	20.0	72.4	mean	6.5	23.7	69.8
industrial areas (IA)				traffic areas (TA)			
Maximum	15.4	36.1	77.6	maximum	14.3	35.4	76.7
Minimum	3.1	19.2	48.5	minimum	4.5	18.4	50.3
Mean	8.7	26.8	64.5	mean	6.7	24.7	68.6
garden areas (GA)				housing areas (HA)			
Maximum	15.7	33.9	81.3	maximum	12.3	33.8	82.5
Minimum	7.8	10.9	50.4	minimum	4.0	13.5	53.9
Mean	9.9	22.7	67.4	mean	6.0	19.4	74.6

The Se levels in oxidised organic-bound forms were high in soils and amounted to 40.1% (Table 5). The highest values were observed in the topsoils of the industrial and allotment garden areas. In virtually each of the analysed profiles, a slight increase in the Se concentration was noted in the illuviation horizon. However, it ought to be mentioned that the extracted $K_2S_2O_8$ fraction manifested the highest stability in content fluctuations of the analysed profiles.

In the current study, the residual fraction was dominant, at 48.5% up to 92.7%. The lowest amounts of this fraction were found in the eluvial horizon, while the highest ones were in the topsoil and the illuvial horizon. This distribution could indicate some strong interaction between Se and soil minerals, including metal oxides, such as Fe, Al and Mn, as confirmed by Wang and Chen (2003) and other authors (Hamdy, Gissel-Nielsen 1970, Johnsson 1991, Tolu et al. 2011).

CONCLUSIONS

1. Sequential extraction of Se from soil subtantiated an evaluation of its behaviour in lessive urban soils of different use and degree of anthropogenic transformation. The analysis of factors determining Se immobilisation or mobilisation in the Lublin area soils demonstrated that the function of land in areas of diverse land use and anthropogenic pressure has an impact on the Se content in elemental and other forms. The soil profile located in the industrial area (IA) and in the allotment garden area (GA) manifested the highest Se content.

2. The distribution of Se concentrations in soil profiles demonstrated a tendency towards the highest Se values in the topsoil, decreasing with depth. Moreover, it was shown that exchangeable and oxidised organic-bound forms correlate with the organic and mineral content of the 1-0.1 mm fraction. Furthermore, no correlation between the selected Se forms and the silt soil fraction was observed, whereas a negative correlation was noted with the fine clay fraction.

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