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ANALYSIS OF THE RELATIONSHIP BETWEEN THE CONTENT OF HEAVY METALS IN SOIL AND DUST IN COPŞA MICĂ

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ABSTRACT: The town of Copşa Mică is polluted with heavy metals due to the metallurgical plant that operated there. Even if the activities of the plant were drastically reduced and depollution equipment was installed, there were high levels of heavy metals in the soil, with long remanence. The major human exposure pathways of heavy metals in the soil are by ingestion of soil particles, by ingestion of home-produced vegetables and by inhalation of dust. This research investigated the levels of heavy metals in the soil, in outdoor and indoor dust, and the relationship between them. The statistical analysis through simple linear regression and correlation showed a statistically significant relationship between the level of heavy metals in the soil (independent variable) and the level of heavy metals in outdoor dust (dependent variable). For Pb and Zn, the linear regression model showed that in areas where high levels of heavy metals are recorded in the soil, high levels of heavy metals are expected in the outdoor dust. Distribution maps of heavy metals in soil and dust also highlight this relationship.

Key words: *soil, dust, heavy metals, Copșa Mică.*

INTRODUCTION

The town of Copșa Mică is polluted with heavy metals because of the metallurgical plant that operated there (Lăcătuşu, 2014, Szanto et al., 2012, A.P. Sibiu, 2010). Heavy metals have a long persistence in the soil, between tens and thousands of years (Kabata-Pendias, 2001), so the pollution and exposure problems of the population are present.

In Copșa Mică, the population health is affected by heavy metals in soil and dust, the main exposure pathways being by ingestion of soil particles, by inhalation of dust and by consumption of home-produced vegetables (Gurzău et al., 2010a, 2010b). The first cases of exposure to Pb were highlighted by Bardac (1999) and Comănescu et al. (2010), which showed that the occupational exposure of employees caused the occurrence of numerous cases of saturnism. Subsequently, research has been carried out to assess exposure to Pb of children (Gurzău et al., 2008). There were found high blod lead level (BLL) with values between 12.5-65 μg/dl in 2002, an average of 45 μg/dl in 2003 and an average of 39 μg/dL in 2008.

Even though the metallurgical plant (SOMETRA) has reduced its activity and the atmospheric emissions have decreased significantly since 2009 (A.P. Sibiu, 2010-2018), the high levels of heavy metals in the soil are a permanent source of population exposure.

There are researches showing that soil can become an important source of dust emissions containing heavy metals, under certain conditions of denuded soil, erosion, dryness and wind speed, surface particles being suspended and transported by air streams (Young et al., 2002, Hillel, 2008, Harris et al., 2009). Wind erosion can be an important source of airborne particulate loadings as well as of specific toxic constituents (Sullivan and Ajwa, 2011).

Other research evaluating the heavy metals in soil and dust on children's playgrounds has shown high levels of metals in the vicinity of industrial platforms. It has been concluded that some of the metals in the dust can be associated with the soil base material, while others are associated with atmospheric pollution, being transported by air and deposited on the surfaces.

Research results showed a strong spatial autocorrelation between the levels of heavy metals in dust and soil (Jin et. al, 2019).

In Copșa Mică, because the remanence of heavy metals in the soil is long-term, the soil is a permanent source of population exposure and a source of dust emissions containing heavy metals.

Because in industrial areas, the main human exposure pathways of heavy metals are by ingestion of soil particles and inhalation of dust (Gurzău et al., 2008), cumulated with the fact that under certain conditions the soil is an important source of heavy metals, through the phenomenon of suspension/ resuspension of the particles from its surface, the statistical analysis of the laboratory results was carried out. It has been tested if there is a statistically significant relationship between: 1) the content of heavy metals in the soil and also the content of heavy metals of outdoor dust; 2) the content of heavy metals content of the indoor and outdoor dust. It is important to understand the phenomenon to substantiate future measures in order to reduce the population exposure to heavy metals in soil and dust.

MATERIALS AND METHODS

Sampling methods

The study was conducted in Copșa Mică in 2014, where 20 individual households were investigated. Soil and dust samples were taken from certain functional areas within each household, as follows: 3 soil samples from each household, from the access area, yard and garden; 4 samples of dust from each household, from the access area, from the kitchen floor, bedrooms and from the investigated subjects right hands; one person from each household was investigated. 60 soil samples and 80 dust samples were obtained following the field stage.

Households were established to be evenly distributed in the town, on all directions, relative to the emission source (the flue-stack of the metallurgical unit), being localized as figure 1.

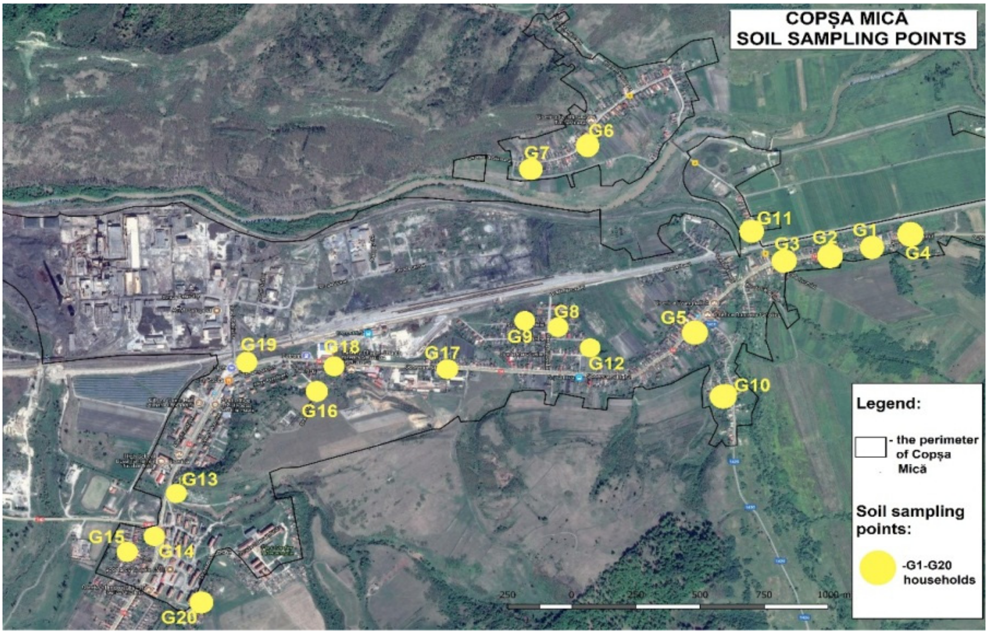

Fig. 1. Copșa Mică, location of investigated households

Because one of the objectives of the research is to establish a relationship between the content of heavy metals in the soil and the content of heavy metals in the dust, soil sampling was carried out from the horizon surface, from a depth of 0-5 cm. Furthermore, previous research has shown the highest concentrations of heavy metals being in the soil surface horizon (Damian et al., 2008).

Dust sampling was taken with dust sampling wipe (LeadWipe) pre-moistened with deionized water, polyorbate 20, methylparaben and propylparaben, used to collect samples of the following surface metals as specified in OSHA Method ID-125G, Addendum B: Pb, Zn, Cu, Cd, Cr, Ni etc. Sampling wipes have the dimensions of 5 x 7.75 inch, meaning 250 cmp, the sampled surface being the surface of the wipe used. Collection procedure complied with OSHA Technical Manual (OTM), Section II, Chapter 2, Apendix C – Procedure for Collecting Wipe Samples.

Soil and dust sample analysis

To determine concentrations of heavy metals in soil (Pb, Cd, Cu, Zn, As), the analyzes were performed in the Environmental Health Center laboratory, Cluj.

Analytical method for heavy metals in soil: X-Ray fluorescence spectrometry (XRF).

Analytical equipment used: Niton™ XL3t XRF Analyzer (Thermo Scientific™).

Method for laboratory analysis used: US EPA Method 6200 reference method.

To determine the level of heavy metals in dust (Pb, Cd, Cu, Zn, As), the samples were analyzed on the same equipment - Niton XL3t 600, by X-ray fluorescence spectrometry method.

Data analysis

Statistical analysis was conducted using Data Analysis tool in Excel. Pearson correlation analysis was conducted to estimate the linear dependence between variables (heavy metals in soil, in outdoor dust and in indoor dust). The correlation coefficient r (Pearson) takes values between -1 and +1, passing through 0, which indicates a null correlation. For the positive interpretation of the correlation coefficient (r) we use: $0 < r \leq 0.1$ - very weak correlation; $0.1 < r \leq 0.39$ - week correlation; $0.4 < r \leq 0.69$ - moderate correlation; $0.7 < r \leq 0.89$ - strong correlation; $0.9 < r \leq 1$ - very strong correlation (Schober et al., 2018).

Spatial distribution of heavy metal levels in soil and dust was performed using the Kriging interpolation method in the ArcGIS, a method which helps determine and assign values to missing points based on measured values in their vicinity.

After analyzing the spatial distribution maps, similarities were observed in the distribution of heavy metals in soil and dust, so advanced statistical methods were used to verify the relationships between them. Statistical data processing was performed using the simple linear regression model.

Linear regression represents a mathematical relationship between an independent variable and a dependent variable. In this research, the relationship between the heavy metal levels in the soil, as an independent variable, and the levels of heavy metals in the outdoor dust, as a dependent variable, was tested and the results were interpreted in terms of statistical significance. It has been assumed that under certain conditions the soil becomes a source of heavy metals, by suspending the particles from its surface, possibly with a relationship between the level of heavy metals in the soil and the level of heavy metals in the dust.

Two other sets of data were tested, one representing the level of heavy metals in outdoor dust, as an independent variable and the level of heavy metals in indoor dust, as a dependent variable. It has been assumed that indoor dust comes partly from the outside of households, which is carried by air, footwear, clothing and other objects.

The ANOVA test was applied to verify the significance of regression and it shows that the model is relevant if the p (*Sig. F*) value <0.05, at a confidence level of 95%.

Before regression testing, the scatter-plot was used to bring information about the two data series and verify if there is a relationship between them.

RESULTS AND DISCUSSIONS

Analysis of heavy metal in soil

The results of the analyzes performed were centralized into a .xlsx document and were interpreted through summary statistical methods.

The results of the analyzes showed that average values of concentrations were recorded in decreasing order **Zn > Pb > Cu > As > Cd**, with the following values (mg/kgSU): Zn – 2350.23; Pb – 1307.85; Cu – 196.29; As – 54.94; Cd – 32.88.

Table 1. Values of minimum, mean and maximum concentrations for lead (Pb) and arsenic (As) in soil, in different function areas (mg/kgSU)

Sampling area	Pb			As		
	min	med	max	min	med	max
Access area (street)	157.73	1550.22	8141.12	16.85	63.06	302.51
Yard	214.98	1465.79	4496.61	12.97	62.84	152.29
Garden	270.54	**907.55**	2595.45	20.43	**36.93**	69.39

Table 2. Values of the minimum, mean and maximum concentrations for cadmium (Cd), copper (Cu) and zinc (Zn) in soil, in different function areas (mg/kgSU)

Sampling area	Cd			Cu			Zn		
	min	med	max	min	med	max	min	med	max
Access area (street)	9.54	46.26	236.90	57.68	232.91	1123.46	543.75	2843.85	17661.06
Yard	11.33	32.81	84.84	56.19	224.59	1373.17	531.21	2566.05	8221.26
Garden	8.84	**21.82**	53.28	61.76	**131.37**	233.87	610.00	**1640.81**	3599.07

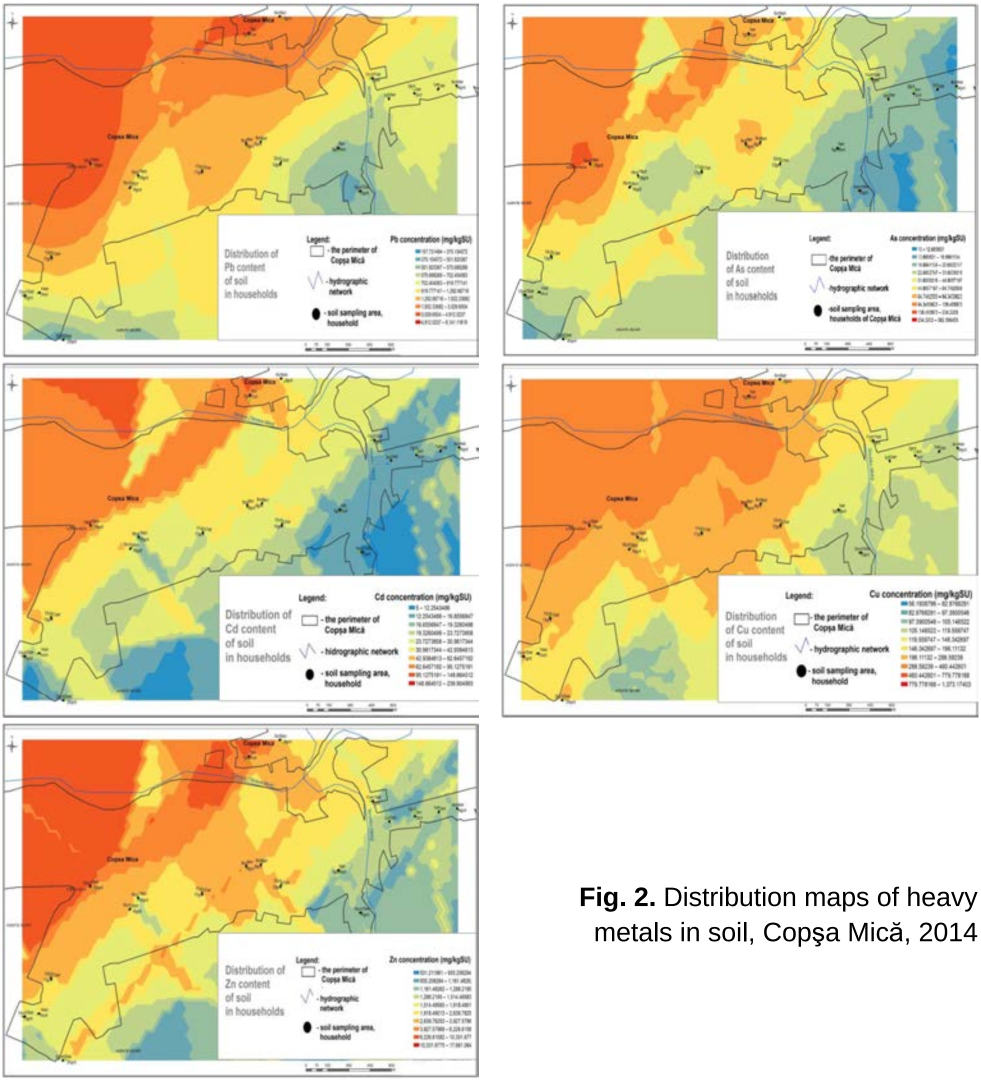

Fig. 2. Distribution maps of heavy metals in soil, Copşa Mică, 2014

Without exception, the average concentrations of heavy metals in the soil were recorded in descending order: **in the access area (street) > in the yard > in the garden**.

The spatial distribution of heavy metals concentrations in the soil of Copşa Mică highlights the areas most affected by pollution in relation to the emission source (the flue-stack of the metallurgical unit).

The distribution maps for heavy metals in the soil show a similarity between them in terms of peak concentrations, the highest values being recorded in 3 zones: at S of the industrial platform; in the Târnăvioara neighborhood, to NE of the industrial platform; in the E-SE neighborhood of the industrial platform. These are the areas most affected by pollution.

Correlation test for heavy metal in soil

Using the DataAnalysis tool in Excel, correlation matrix was accomplished.

Table 3. Correlation matrix for heavy metals in soil, Copşa Mică, 2014

	Pb	**As**	**Cd**	**Cu**	**Zn**
Pb	1				
As	0.856681	1			
Cd	0.962907	0.87753	1		
Cu	0.63614	0.770931	0.664759	1	
Zn	0.928036	0.940251	0.945554	0.835631	1

There is a very strong correlation between the concentrations of heavy metals for Pb-Cd, Pb-Zn, Zn-As, Zn-Cd, a strong correlation between As-Pb, As-Cd, As-Cu and a moderate correlation between Cu-Pb, Cu-Cd.

The correlation between these five metals is not by accident, and they are influenced by the same soil pollution phenomenon that has been present for decades in Copșa Mică.

Analysis of heavy metal in dust

Following the laboratory analysis, values below the method detection limit (< LOD) were recorded for As and Cd.

The results of the analyzes for Pb, Cu and Zn in dust, were centralized into a. xlsx document and were interpreted through summary statistical methods.

Table 4. Values of minimum, mean and maximum levels for lead (Pb), cooper (Cu) and zinc (Zn) in dust, in different function areas (μg/cm^2)

Sampling area	**Pb**			**Zn**			**Cu**		
	min	**med**	**max**	**min**	**med**	**max**	**min**	**med**	**max**
Acces area (yard)	0.0473	0.2160	0.5774	0.1319	0.2552	0.3857	0.0252	0.0395	0.0481
Chicken	0.0451	0.0780	0.1359	0.1069	0.1850	0.3163	0.0329	0.0400	0.0566
Bedroom	0.0397	0.1033	0.4576	0.1278	0.1943	0.3927	0.0313	0.0387	0.0527
Right hand	0.0607	0.1306	0.2672	0.1124	0.1692	0.3234	0.0340	0.0449	0.0878

The three metals analyzed from the dust have recorded average levels in decreasing order: **Zn> Pb> Cu**, as follows (μg/cm^2): Zn – 0.2009; Pb – 0.1598; Cu – 0.0408.

Comparing the average levels of heavy metals in dust, with the average concentrations of heavy metals in the soil, it can be observed that the highest average in soil was also recorded for Zn in descending order: **Zn> Pb> Cu**> As> Cd.

Unlike the concentrations of heavy metals in soil, where there is an order in functional areas, with the highest average of the access (street) and the lowest average in the gardens, there is no order for dust.

The highest average content of Pb and Zn in the dust were recorded outside the household area, while for Cu the highest average being recorded on the right hand of the investigated subjects.

The distribution maps for the 3 metals in the dust of Copșa Mică show a correlation regarding the highest levels in the SE of the town (figure 3). This area does not overlap areas with high concentrations of heavy metals in the soil.

For Pb and Zn, the highest values are in the NE – Târnăvioara neighborhood and in the vicinity of the industrial platform, in S of it, similar to the heavy metal distribution in the soil.

A relation to the spatial distribution of Pb and Zn from dust can be observed.

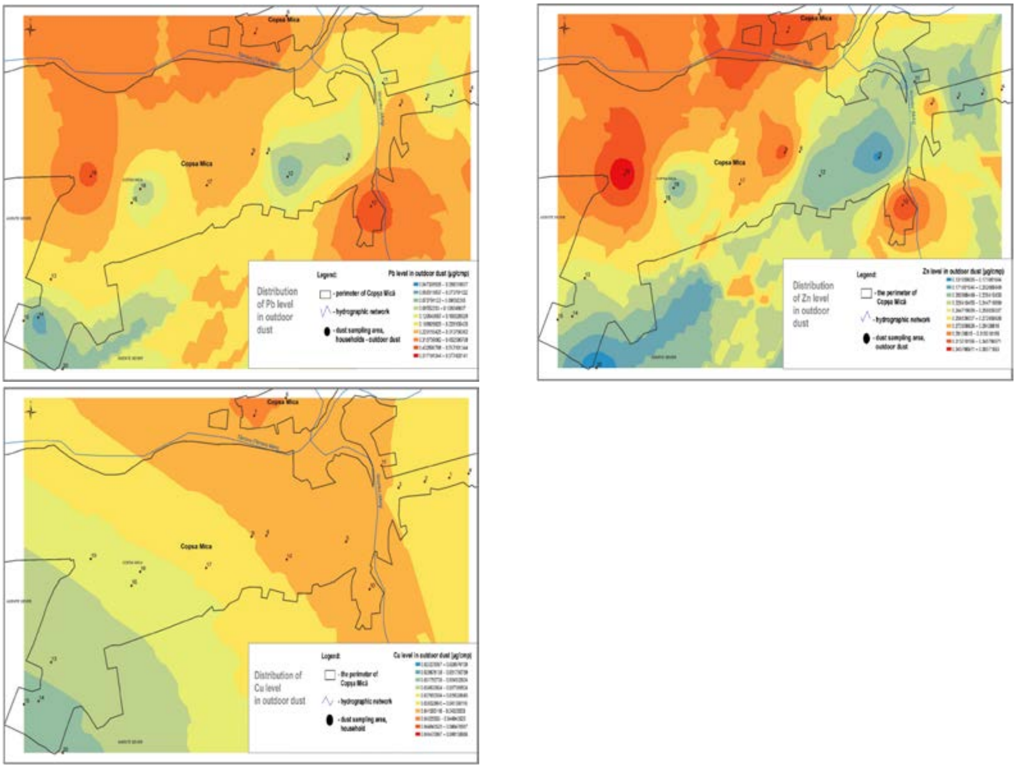

Fig. 3. Distribution maps of heavy metals in outdoor dust, Copşa Mică, 2014

Correlation test for heavy metal in dust

Using the DataAnalysis tool in Excel, correlation matrix was obtained.

Table 5. Correlation matrix for heavy metals in dust, Copşa Mică, 2014

	Pb	**Cu**	**Zn**
Pb	1		
Cu	0.298078	1	
Zn	0.822298	0.250295	1

As observed on the distribution maps, there is a strong correlation between the Pb-Zn levels in the dust and a poor correlation between Cu-Pb and Cu-Zn. The lowest values of the correlation coefficient are for Cu with the other metals, noticed also on the distribution maps. Similarly, in the correlation test for heavy metals concentrations in the soil, Cu recorded the lowest values of the correlation coefficient in relation to the other metals.

Statistical analysis of heavy metals in soil and in outdoor dust

Scattering diagrams for the data pairs have been developed: concentrations of heavy metals in soil – levels of heavy metals in dust. Because the As and Cd levels in the dust were below the method detection limit, the scattering and statistical regression analysis were performed only for Pb, Zn and Cu.

For each household, the average concentration of heavy metals in the soil was calculated in all three functional areas: from the access to the household, yard and garden. The obtained data series, represented by the average concentrations of Pb, Zn and Cu in the soil for each household, was considered as the independent variable, and the level of metals in dust from outdoor was considered the dependent variable.

Lead in soil and outdoor dust

According to the cloud of points form, a relationship between the level of Pb in the soil of households and the Pb level in outdoor dust is considered probable (figure 4).

The value of the correlation coefficient R^2 shows that the dependent variable (Pb in the dust) is explained in a proportion of 33.45% by the regression equation, i.e the independent variable (Pb in the soil). The linear regression model is defined by the equation given on the regression line.

The correlation test performed with the Data Analysis tool in Excel shows that there is a moderate correlation between the Pb level in the soil of households and the Pb level in outdoor dust (r = 0.57).

The statistics of correlation coefficient R^2 with the value of 0.334 shows that the model, as tested, justifies 33.4% of the variability of Pb levels in outdoor dust in relation to Pb level in the soil.

In the ANOVA test, p value (*Sig. F*) is less than 0.05, so the hypothesis of the lack of significance of the independent variable is rejected, in favor of the hypothesis that the regression model is statistically significant.

Coefficients in the regression equation are significant because p values are less than 0.05 ($p_{intercept}$ = 0.04 <0.05; $p_{Pb\text{-}soil}$ = 0.007 < 0.05), and the confidence intervals for the 2 coefficients do not contain the zero value, so the proposed model is statistically significant at a confidence level of 95%.

The regression equation that defines the model, resulted from the test and from the scattering diagram: y = 8E-05x + 0.1054; the coefficients of the equation are statistically significant.

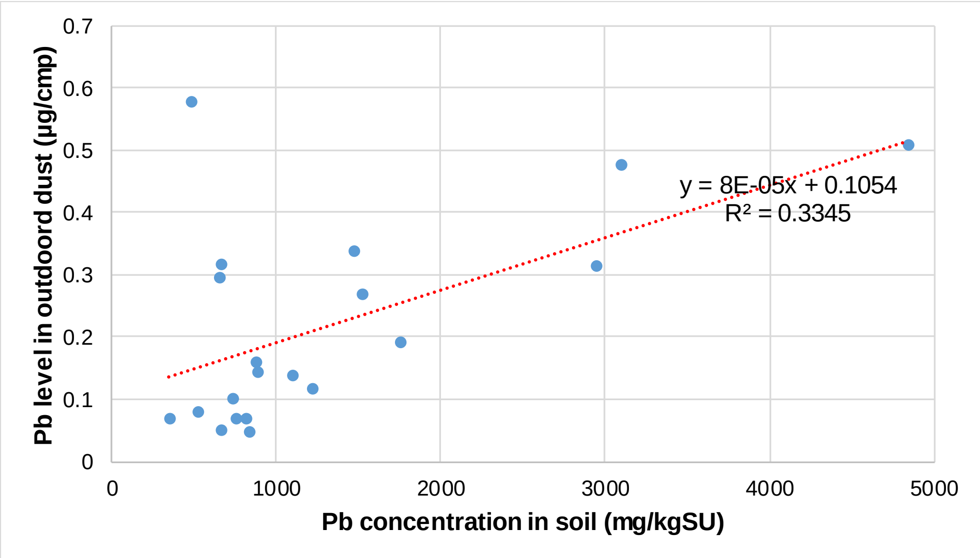

Fig. 4. Scattering diagram for Pb concentration in soil and Pb level in outdoor dust

Table 6. Correlation matrix for Pb in soil and in outdoor dust, Copşa Mică, 2014

	Pb-soil	Pb-dust yard
Pb-soil	1	
Pb-dust yard	**0.5783943**	1

The ANOVA test results and the linear regression coefficients obtained are presented.

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.5783943
R Square	0.3345399
Adjusted R Square	0.2975699
Standard Error	0.1367547
Observations	20

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.1692322	0.169232232	9.048955139	0.00755
Residual	18	0.3366334	0.018701853		
Total	19	0.5058656			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	0.1054006	0.0478379	2.203285049	0.040840286	0.004897	0.205904
Pb-soil	8.462E-05	2.813E-05	3.008148124	0.007550401	2.55E-05	0.000144

Zinc in soil and in outdoor dust

According to the cloud of points form, a relationship between the level of Zn in the soil of households and the level of Zn in outdoor dust is considered probable (figure 5).

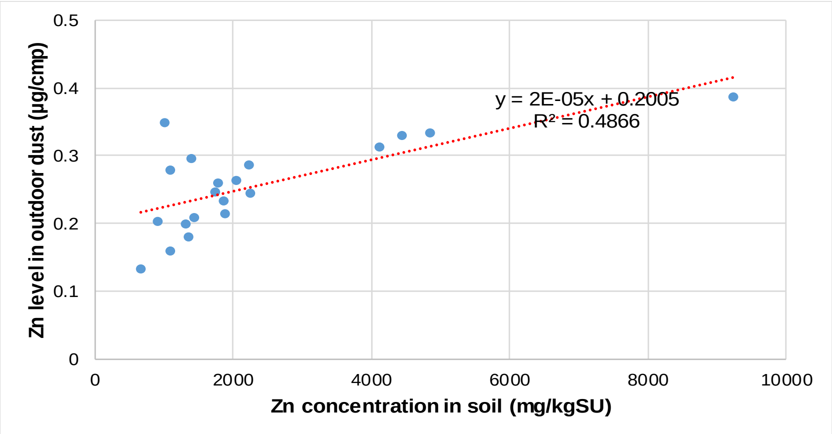

Fig. 5. Scattering diagram for the concentration of Zn in soil and Zn level in outdoor dust

The value of the correlation coefficient R^2 shows that the dependent variable (Zn in dust) is explained in a proportion of 48.66% by the regression equation, i.e the independent variable (Zn in soil). The linear regression model is defined by the equation given on the regression line.

The correlation test performed with the Data Analysis tool in Excel shows that there is a moderate to strong correlation between the Zn level in the soil of households and the level of Zn in outdoor dust (r = 0.697).

Table 7. Correlation matrix for Zn in soil and in outdoor dust, Copşa Mică, 2014

	Zn-soil	Zn-dust yard
Zn-soil	1	
Zn-dust yard	**0.697586**	1

The testing of the linear regression model, with ANOVA, is presented below.

Statistics of correlation coefficient R^2 with the value of 0.4866 shows that the model, as tested, justifies 48.66% of the variability of Zn levels in the outdoor dust in relation to the level of Zn in the soil.

In the ANOVA test, p value (*Sig. F*) is less than 0.05, so the hypothesis of the lack of significance of the independent variable is rejected, in favor of the hypothesis that the regression model is statistically significant.

Coefficients in the regression equation are significant because the p values are less than 0.05 ($p_{intercept} < 0.05$; $p_{Zn\text{-}soil} < 0.05$), and the confidence intervals for the 2 coefficients do not contain the zero value, so the proposed model is statistically significant at a confidence level of 95%.

The regression equation that defines the model resulted from the test and from the scattering diagram (y = 2E-05x + 0.2005); the coefficients of the equation are statistically significant.

Statistical testing for the linear regression model showed that it is not significant for the relationship between Cu in soil and Cu in outdoor dust.

Statistical tests performed on heavy metals (Pb, Zn, Cu) in outdoor dust and indoor dust, or from the hands of the investigated subjects, did not show a statistically significant relationship. The influence of households cleaning activities and personal hygiene (hand washing) is an important one and significantly affects the level of heavy metals in indoor dust and on hands.

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.697586
R Square	0.486626
Adjusted R Square	0.458105
Standard Error	0.049087
Observations	20

ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.041111	0.041111138	17.06217499	0.00062793
Residual	18	0.043371	0.00240949		
Total	19	0.084482			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.200549	0.017205	11.65661778	8.04173E-10	0.16440348	0.236695206
Zn-soil	2.33E-05	5.64E-06	4.130638569	0.000627934	1.1442E-05	3.51286E-05

CONCLUSIONS

For the analysis of heavy metals in the soil, the results showed that that average values of concentrations were recorded in decreasing order **Zn > Pb > Cu > As > Cd**, with the following values (mg/kgSU): Zn – 2350.23; Pb – 1307.85; Cu – 196.29; As – 54.94; Cd – 32.88.

Without exception, the average concentrations of heavy metals in the soil were recorded in descending order **in the access area (street) > in the yard > in the garden.**

The distribution maps for heavy metals in the soil show a similarity in their distribution, the highest values being recorded in three areas: at S of the industrial platform; in the Târnăvioara neighborhood, to NE of the industrial platform; in the E-SE neighborhood of the industrial platform.

The correlation test for heavy metals in soil shows that there is a very strong correlation between the concentrations of heavy metals for Pb-Cd, Pb-Zn, Zn-As, Zn-Cd, a strong correlation between As-Pb, As-Cd, As-Cu and a moderate correlation between Cu-Pb, Cu-Cd.

The correlation between these 5 metals is not a random, and they are influenced by the same soil pollution phenomenon that has been present for decades in Copșa Mică. For Cu the lowest correlation indices have been recorded, besides the pollution phenomenon, probably coming from the natural background.

After analyzing the levels of heavy metals in outdoor and indoor dust, it was found that levels of Cd and As in the dust were below the method detection limit.

The three metals (Pb, Cu, Zn) in dust recorded average levels in a decreasing order **Zn > Pb > Cu**, with the following values (μg/cm^2): Zn – 0.2009; Pb – 0.1598; Cu – 0.0408. The decreasing level of average for heavy metals in the dust is similar to the decreasing order of the metals concentrations in the soil (**Zn > Pb > Cu** > As > Cd).

The highest averages for the content of Pb and Zn in dust were recorded outdoor; for Cu the highest average is recorded on the right hand of the investigated subjects.

Unlike heavy metal concentrations in the soil where a rule has been observed on functional areas, for heavy metals in the dust there is no rule. The cause may be that the level of heavy metals in the functional areas depends on the frequency and proper cleaning (dry/wet), and in the hands case depends on hands wash behavior.

According to the distribution maps for heavy metals in dust, the highest levels of Pb and Zn in outdoor dust were recorded in the vicinity of the industrial platform, at S; in NE, in the Târnăvioara neighborhood; in SE, at the exit towards Valea Viilor and the E-SE neighborhood of the industrial platform. The three areas with the highest levels of heavy metals in the dust overlap the areas with high levels of heavy metals in the soil.

The results of the correlation test for heavy metals in dust show a strong correlation between Pb-Zn and a poor correlation between Cu-Pb and Cu-Zn. The lowest values of the correlation coefficient are for Cu with the other metals in the dust, as well as for the soil.

Statistical analysis through regression and correlation did not reveal a statistically significant relationship between the levels of heavy metals in outdoor and indoor dust or on hands of investigated subjects. Proper households cleaning and hand washing behavior significantly influence the level of heavy metals in indoor dust and also dust on hands.

For Pb and Zn, the linear regression model highlights that in areas were high levels of heavy metals in soil are expected, linked to them, high levels of heavy metals in dust are expected. This has also been highlighted on the distribution maps of heavy metals in soil and on the distribution maps of heavy metals in dust.

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