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STUDY OF THE GROUNDWATER POLLUTION WITH SULPHATES AND LEAD IN THE INDUSTRIAL AREA OF ORADEA MUNICIPALITY, ROMANIA

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Abstract

The aim of the study was to determine the evolution of sulphates and lead concentrations in groundwater on the western area of Oradea (Romania). The groundwater is stored in Crisul Repede alluvial cone which is developed on both banks and the body of groundwater ROCR 01 is the source of drinking water supply of Oradea. In the 1960s was developed an industrial platform and after 1989, there were essential changes in the industrial economic structure. The water quality has been analyzed for a period of 40 years, starting with 1977. In order to monitor the evolution of the two studied parameters were used the data obtained by the Control Station of Industrial Pollution (CSIP) in the industrial zone, provided by the "Crişuri" Water Administration Basin. Based on this pre-existing database was studied the variation of the sulphates and lead concentrations and was observed that the surfaces with groundwater polluted with SO4 are reduced in the 40 years of observations by 48.78% and in the case of Pb the reduction is 51.83%.

Key words: *groundwater, industrial pollution, water quality, sulphates, lead.*

INTRODUCTION

Drinking water quality is a matter of ecological and social importance both in the urban environment (Duzen, 2017) and in rural areas. Considering that most of the populated centers use groundwater for water supply, their quality is influenced by the pollution caused by anthropogenic activities.
In the case of urban agglomerations, pollution is due to excessive urbanization and industrialization (Marić et al., 2014) and in the case of rural settlements due to the intensive agriculture (Neziri et al., 2014).
Underground waters are influenced, both quantitatively and qualitatively, by the current global climate change and by its irrational use. Their rational management is a challenge (Tellam et al., 2006), especially in arid and semi-arid areas (Salihou Djari et al., 2018), where the coverage of water needs to feed the population, competes with the water needs of various uses, including the irrigation water, required for agricultural holdings (Gálya et al., 2018; Imbrea et al., 2017). The metallurgical and energy industries pollute the environmental factors through the tailings ponds and tailings dumps resulting from the production process. The uncontrolled landfill leachates of household waste in areas near major urban agglomerations is another important source of contamination of groundwater and surface water (Rusu et al., 2017; Şmuleac et al., 2017).
Ispas et al. (2018) analyzing the drinking water quality of the Haneş mining area, Alba County reports high levels of sulfur in all analyzed samples in the years 2013-2015 and highlights the risk of groundwater pollution with sulphates (SO4).
Schmidt et al. (2017), following the study on the risk assessment of pollution with toxic elements of groundwater from Slovakia and the Bratislava region, points out that, although polluted surfaces with heavy metals are small, the risk to human health should not be

underestimated, especially in the intensive populated areas.
The studies were conducted in Oradea, Bihor County, located in the northwest part of Romania. In this paper was studied the influence of the industrial activities restructuring in the industrial zone of Oradea on the temporal evolution of sulphates (SO_4) and lead (Pb) in groundwater. From the 1960s until 1985, in the western part of Oradea, on the right bank of Crişul Repede, the industrial platform with an area of approximately 9 km^2 was developed (Romocea et al., 2018). The main industrial enterprises that operate here are: Thermoelectric Power Plant (TPP), "Alumina" Metallurgical Plant (AMP), "Sinteza" Chemical Factory (SCF), "UAMT" Automotive Metalworking (UAM), "Diamant" Sugar Factory (DSF), "Alfa" Woodworking and Furniture Production (AWFP), Construction Materials Industry (careers for ballast operation) and Waste Water Treatment Plant (WWTP). Since 2004 in this area, there is a controlled landfill leachates of household waste (LLHW), managed by S.C. Eco BIHOR S.R.L., located near the uncontrolled landfill leachates of household waste, which was closed.
Forced by necessity to implement EU environmental legislation (DIRECTIVE 91/271/EEC, 1991; DIRECTIVE 118/EC, 2006; ORDER ME no. 137, 2009) the plants and factories with a major negative impact on the water quality, which were active in the area, have greatly restricted their activity, restructured their production or went bankrupt. The industrial zone of Oradea has shown interest in assessing the impact of industrial activities on the main environmental factors, reported by: Romocea et al. (2003), impact of SCF on groundwater; Man et al. (2003), influence of tailings dumps from TPP Oradea on groundwater; Orlescu et al. (2003), influence of the tailings ponds from AMP Oradea on environmental factors.

MATERIALS AND METHODS

To study the temporal evolution of sulphates and lead in groundwater, cantonated in the water body ROCR 01 were used the data obtained by the Control Station of Industrial Pollution (CSIP) in the industrial zone, for a period of 40 years, provided by the "Crişuri" Water Administration Basin (Narw, 2017). For the study were used the years with complete analyzes (1977, 1986, 1990, which is representative of the industrial development of the communist period, 1995, 1997, 2008, 2010 and 2017, respectively). Entire network of the hydrogeological drillings consist of 11 hydrogeological drillings but for the study were selected 9 drillings on the right side of the watercourse.
These are relevant through their location near industrial sites and through the complete set of analyzes. 4 drillings (F1, F2, F6-P13 and F7-P14) are part of the first-order hydrogeological crossing, located in the south-north direction across the Crişul Repede, to which the second-order drilling (F1-P15) was added in time. This is located at Sântăul Mic, near the tailing pond of TPP Oradea. The other hydrogeological drillings in the number of 6 (P1, P2, P3, P5, P6, P7) comprise the CSIP of groundwater in the industrial area, being disposed in the east-west direction, approximately parallel to the Oradea-Borş road, in proximity of the main industrial objectives.
The maximum admissible concentrations (MAC) of sulphates and lead according to LAW 458, 2002 and the methods of analysis are presented in Table 1. To analyse the statistical significance of the trends was used ANOVA variance analysis for a factor and for two factors. To test the possible correlations between the analyzed qualitative parameter values and the temporal and spatial characteristics the GNU PSPP Statistical Analysis Software was used (GNU PSPP, 2016).
The spatial trends of the analyzed parameters was highlighted with the GIS software, ESRI ArcGIS 9.3. In order to cover the area with agricultural uses the analyzed perimeter area was increased with 9 km^2 to the west, resulting an area of 22.721 km^2.

Table 1. The MAC for analyzed parameter and the analysis methods

Categories	Sulphates SO_4^{2-}	Lead Pb
U.M.	mg/l	mg/l
MAC	250	0.01
Analyses methods	STAS 8601/70	STAS 6362/85

RESULTS AND DISCUSSIONS

The groundwater from the studied hydrogeological drillings is cantonated in the water body ROCR 01 which is a cross-border underground water reservoir, developed in the alluvial cone of Crișul Repede. In the industrial area of Oradea municipality, it has depths between 2.00 m and 8.00 m and thicknesses of water-bearing horizon of 10-17 m. In the industrial area, the underground waters are drained, to the south by the natural course of Crişul Repede and to the north by the tributary Crisul Mic river, that flowing in the east - northwest direction and passing through the territory of Hungary to the west of Santăul Mare.

The underground water of this body is fed by rainfall and leakage from the slopes of Oradea Hills, located to the east of the analyzed area and to large waters, through infiltrations from the Crişul Repede River.

In the analyzed perimeter, Orlescu et al. (2003) signals the possibility of SO_4 pollution, due to sludge deposits from AMP and Man et al. (2003) mentions the TPP Oradea tailings pond as possible source of groundwater pollution. The evolution of SO_4 in the Oradea industrial zone is aimed at identifying the periods with exceedances of the MAC of 250 mg/l and the polluted surfaces.

The statistical analysis of the SO_4 concentration values, in the waters collected from the hydrogeological drilling from the area, during the period 1977-2017 shows that multiannual averages exceed MAC in four cases (P1, P6, F6 and F7) The standard error of the mean is between 6.54 mg/l and 39.42 mg/l. The Kurtosis values (K) show that the error distribution closest to the normal distribution curve is encountered at the P3 drilling (K = 0.95 and the distribution curve displaced in the negative area of the errors is specific to the F7 drilling, with the highest negative K value, K = -1.57 (Table 2).

Annual mean of SO_4 concentrations are the most common exceedances of MAC at drills: F6 drilling in 7 cases (1977, 1986, 1990, 2008, 2010 and 2017); P1 in 6 cases (since 1990); P6 in 4 cases (1977, 1995, 2008 and 2010); F7 drilling in 4 cases (1977, 1990, 1995 and 1997). It is noted that, in these points, most cases exceeding the MAC are present after 1997 (Table 3).

The SO_4 concentration increases in the water samples collected from the P1 drilling after 1990 are due to the fact that, upstream of this point, in the direction of the groundwater drainage, (northeast) at Santăul Mic, the tailings pond of TPP came into operation. For the F6 and P6 drillings, located in the central area of the analyzed perimeter, the identification of the polluting enterprises is relative, due to the fact that there are several industrial objectives in their vicinity: TPP Oradea, AMP, DSF and SCF. In the north-east, upstream of them on the direction of groundwater flow, before, 1980 there was a natural lake that was clogged with ashes and sludge from AMP and TPP Oradea. The F7 drill is located in the northeastern part of this lake, currently clogged.

Table 2. Statistical analysis of SO^-_4 concentrations (mg/l) in the groundwater from industrial area Oradea (1977-2017)

Dril	N	Minimum	Maximum	Mean	Standard Error of mean	Standard Deviation	Kurtosis (K)
P1	28	75.05	674.65	317.81	33.48	177.17	-0.89
P3	26	54.48	380.00	178.57	14.44	73.61	0.95
P5	26	49.45	264.60	155.30	10.89	55.52	-0.35
P6	26	74.00	716.43	265.12	31.84	162.36	0.53
P7	26	9.05	232.27	114.78	13.57	69.21	-1.45
P9	24	7.75	126.63	48.34	6.93	33.93	-0.19
F5	26	34.00	141.60	70.38	6.54	33.36	-0.30
F6	28	26.72	640.00	336.26	37.36	197.70	-1.29
F7	26	80.00	649.59	362.95	39.42	201.00	-1.57

Table 3. Evolution of SO^-_4 concentrations (mg/l) in the groundwater from industrial area Oradea (1977-2017)

	P1	P3	P5	P6	P7	P9	F5	F6	F7
MAC	250	250	250	250	250	250	250	250	250
1977	103.15	77.09	67.14	402.32	135.13	97.94	112.11	415.96	637.91
Differ.	- 146.85	- 172.91	- 182.86	152.32	- 114.88	- 152.06	- 137.90	165.96	387.91
1986	154.00	144.50	134.00	114.50	58.00	22.50	59.50	282.25	125.50
Differ.	-96.00	- 105.50	- 116.00	- 135.50	- 192.00	- 227.50	- 190.50	32.25	- 124.50
1990	518.25	268.25	187.50	102.00	135.00	55.25	61.00	525.50	480.00
Differ.	268.25	18.25	-62.50	- 148.00	- 115.00	- 194.75	- 189.00	275.50	230.00
1995	418.10	254.26	218.90	278.48	186.44	19.65	55.90	37.55	468.75
Differ.	168.10	4.26	-31.10	28.48	-63.57	- 230.35	- 194.11	- 212.45	218.75
1997	320.93	204.53	191.90	203.72	158.73	31.27	56.08	118.03	424.29
Differ.	70.93	-45.48	-58.10	-46.28	-91.27	- 218.74	- 193.92	- 131.97	174.29
2008	387.00	168.00	130.89	442.00	26.00	63.45	42.83	490.00	147.00
Differ.	137.00	-82.00	- 119.11	192.00	- 224.00	- 186.55	- 207.17	240.00	- 103.00
2010	351.40	145.10	140.05	360.90	93.50	-	140.05	500.80	151.40
Differ.	101.40	- 104.90	- 109.95	110.90	- 156.50	-	- 109.95	250.80	-98.60
2017	295.10	-	-	-	-	-	-	415.45	-
Differ.	45.1	-	-	-	-	-	-	165.45	-

Correlation of SO_4 concentration - time. The testing of the correlative links between the SO_4 concentrations recorded during the analyzed period, highlights the existence of statistically assured correlative links for all the drills, with the exception of F6 and P7. The closest correlation, of the second degree polynomial type, very statistically significant is found at the P5 drilling, the correlation coefficient being R^2 = 0.7115. The correlation SO_4 concentrations (mg/l) - time (years) in the P1 observation drilling, for 28 pairs of values, situated to the west of the area analyzed, statistically significant (R^2 = 0.3446) shows a peak in the period 1995-2005 (Figure 1). This period of SO_4 maximum concentrations occurred after the closure and safety of the old tailing pond from TPP and AMP and the opening of the new tailing pond from Sântăul Mic. The tendency to reduce SO4 concentrations, after 2005 is explained by the groundwater protection measures, imposed in the exploitation of the Sântăul Mic tailing pond. Evolution of SO_4 concentrations recorded at P6 drilling, located in the central area of the analyzed perimeter, according to time (years) using 26 pairs of values, statistically significant (R^2 = 0.5991) is of polynomial type, grade III (Figure 1). The trend highlighted by the shape of the resulting curve indicates that from the first year of analysis (1977) the determined concentrations are reduced to 1987-1990 when they fall into the MAC, then to increase, except in the last year of observations 2010. The high values of SO_4 concentrations in P6 drilling water, from 2008, could be explained by the deposition of ash from TPP and sludge from the AMP in the former natural lake, located upstream in the direction of the groundwater drainage. After the clogging of the lake mentioned above, the tailing pond located near the Oradea-Borș road, in the immediate vicinity of the drilling was opened. To these are added the cumulative effect of emissions from the industrial enterprises (SCF, TPP and AMP) located in the northeastern neighborhood of P6 drilling.

The tendency to reduce SO_4 concentrations after 2010 can be attributed to the reduction of industrial production from SCF and AMP and respectively to the modernization of TPP by switching from energy production using solid fuels (coal) to natural gas.

The space evolution Analysis of the spatial distribution of SO_4 polluted areas, using ArcGIS 9.3. allows determination of the evolution of unpolluted land areas (with concentrations less than MAC). These increase from 8.788 km^2 in 1977 to 20.458 km^2 in the last year of observations 2017 (Table 4). Surfaces with double MAC overflows are high in 1977, 19.56%, for 1990 to become very low, of 1.496%. In 2010, an area of only 0.032 km^2 (0.141%) for which the SO_4 concentration in groundwater exceeds 2 x MAC is registered.

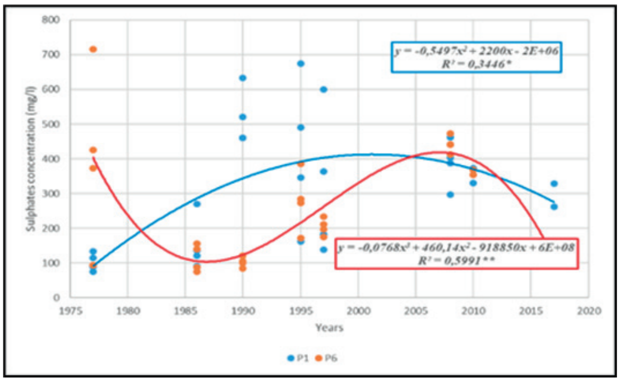

Figure 1. Evolution of SO_4 concentration at P1 and P6 drillings in the analyzed period

Table 4. Surfaces contaminated with SO_4^- (mg/l) in the Oradea industrial area

Year	U.M.	> 2 x MAC	1-2 MAC	< MAC	Total
1977	km^2	4.445	9.489	8,.788	22.722
	%	19.56	41.76	38.68	100.00
1990	km^2	0.340	13.346	9.036	22.722
	%	1.50	58.73	39.77	100.00
1997	km^2	-	9.653	13.069	22.722
	%	-	42.48	57.52	100.00
2008	km^2	-	8.619	14.103	22.722
	%	-	37.93	62.07	100.00
2010	km^2	0.032	7.873	14.817	22.722
	%	0.14	34.66	65.21	100.00
2017	km^2	-	2.264	20.458	22.722
	%	-	9.96	90.04	100.00

Time influences areas affected annually by SO_4 pollution. The correlation of the second degree polynomial type, the percentage area - time (years) is distinct statistically significant (Figure 2). If, before the reference year 1990, the surface on which the groundwater was polluted with SO_4 represented about 60% of the analyzed area, the restructuring of the industrial enterprises from the area, causes the reduction of these surface to less than 10% in 2017. The Pb is one of the most toxic heavy metals, being encountered under natural conditions at very low concentrations in groundwater, due to the fact that, its compounds are poorly soluble. Pb can reach the groundwater, from the upper horizons of the soil profile, where it accumulates by solubilization, under the conditions of acidic waters and long contact with the polluted soil horizons. The MAC for underground water bodies is 0.01 mg/l.

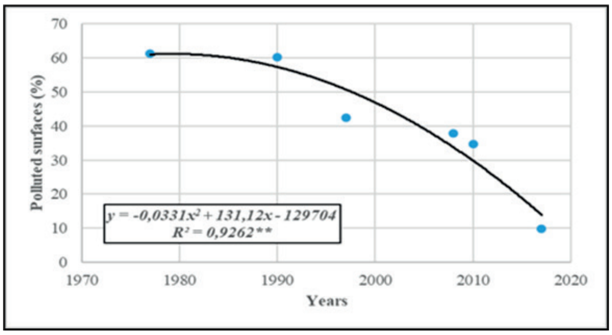

Figure 2. The connection between SO4 polluted areas (%) of and time (years)

The statistical analysis. The mean multiannual values of Pb concentrations, in the water collected from the observation drills are between 0.0067 mg/l and 0.0209 mg/l, with MAC overruns in 6 cases (P1, P3, P5, P6, P7 and P9). All the points of observation with exceedances of the MAC are part of the CSIP being located on one side and the other of the Oradea-Borș road, characterized by intense auto traffic (Table 5).

Table 5. Statistical analysis of Pb concentrations (mg/l) (1977-2017)

Drill	N	Minim	Maxim	Mean	Standard Error of mean	Standard Deviation	Kurtosis (K)
P1	28	0.0002	0.1910	0.0152	0.01	0.04	12.58
P3	26	0.0005	0.0990	0.0111	0.00	0.02	10.66
P5	26	0.0001	0.1550	0.0171	0.01	0.03	12.26
P6	26	0.0003	0.0905	0.0209	0.01	0.03	1.26
P7	26	0.0002	0.0850	0.0147	0.00	0.02	6.95
P9	24	0.0001	0.1230	0.0102	0.01	0.02	21.47
F5	24	0.0001	0.0580	0.0086	0.00	0.01	6.54
F6	28	0.0002	0.0180	0.0067	0.00	0.01	-0.66
F7	26	0.0002	0.0570	0.0075	0.00	0.01	11.51

The maximum values recorded (0.0180-0.1910 mg/l) exceed the MAC for all observation points, but for P1 this exceedance being of 19 times. In the case of 4 observation drillings, Standard Error of Means exceeds MAC (0.01 mg/l), and Standard Deviation is higher than MAC in all analyzed cases. Kurtosis values are very different, presenting in most cases higher frequencies of positive errors, the error distribution curve being far from the normal. The closest error distributions relative to the normal Gaussian curve are found at the hydrogeological wells P6 (K = 1.26) and F6 (K = -0.66) located in the central area of the analyzed surface.

The temporal evolution. The analysis of annual mean values of Pb concentrations in groundwater shows that they exceed the MAC as follows: 6 drillings in the first year of research (P1, P3, P5, P7, F5 and F6); 7 cases in the years 1986 (P1, P5, P6, P7, F5, F6 and F7); 6 in 1990 (P1, P3, P5, P6, P9 and F5); 2 cases in 1995 (P6 and P7); 5 in 1997 (P5, P6, P7, F6 and F7) (Figure 3). In the last three years analyzed all the analyzed drills fall into the MAC. The graphical representation of mean values of Pb concentration in groundwater suggests the general tendency to reduce them with the passage of time.

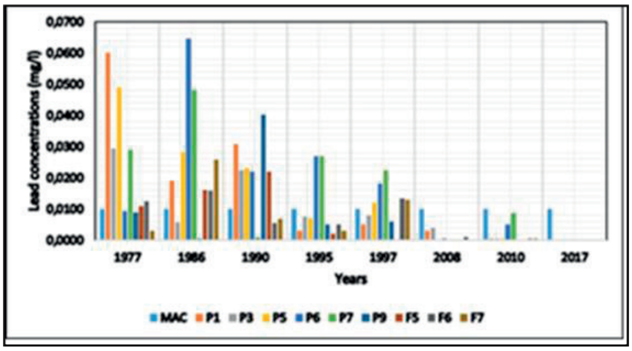

Figure 3. Evolution of annual average Pb concentrations in groundwater (1977-2010)

Correlation of Pb concentration - time. The linear correlations established between the time expressed in years, of the observation period and the Pb concentrations, in the water harvested from the drillings are of exponential type. They show statistical significance only for 4 drillings (P1, P5, F5, F6 and F7) of the 9 analyzed. All drills in the first order cross and only 2 from CSIP have statistically assured correlations.

For the drillings in the first order cross, the closest to Oradea-Borș, F5 and F6, the exponential correlations are very significant (R^2 = 0.6577) and respectively distinctly significant (R^2 = 0.5333) (Figure 4).

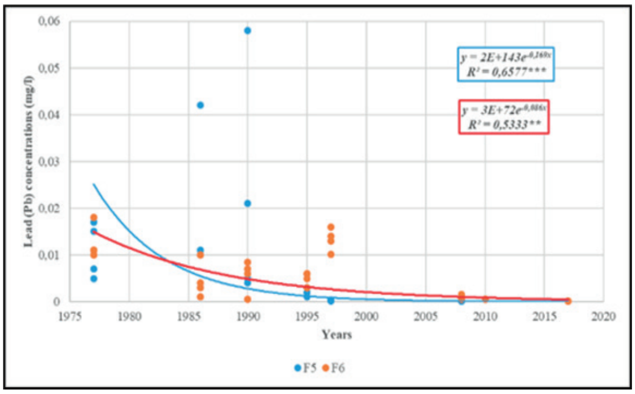

Figure 4. Correlative correlation between Pb concentrations at F5 and F6 and time (years)

The fact that the drillings from the CSIP, located on the vicinity of the road, do not have statistical correlations (except for the P1 and P5 drillings) shows that the main source of contamination with Pb is not the heavy traffic. This source is obvious and manifested itself until the prohibition of Pb-additive gasoline in the 1990s.

The drilling F6, which has the closest correlation, being located near the SCF, suggests that this is the main source of contamination with Pb. It is worth noting that for both observation points the tendency is to reduce Pb concentrations over time, the shape of the curves indicating an annual rate of decrease in concentration, higher at the F5 drilling (0.0008 mg/l per year) located in the vicinity SCF.

The space evolution. Thematic maps obtained using ArcGIS 9.3. confirms that the observation points with the highest concentrations of Pb are located in the central area of the studied perimeter, near the sources of pollution SCF and AMP (F6) and on the direction of drainage of the underground, which passes through this area. By spatial analysis of the Pb concentrations in the groundwater, the areas for which the Pb concentrations exceed 5 times, are between 3-5 MAC and 1-3 MAC were highlighted (Table 6).

Table 6. Polluted surfaces with Pb (mg/l) in the Oradea industrial area

Year	U.M.	> 5 x MAC	3-5 MAC	1-3 MAC	< MAC	Total
1977	km^2	3.524	5.641	5.004	8.553	22.722
	%	15.51	24.83	22.02	37.64	100.00
1990	km^2	-	1.250	11.776	9.696	22.722
	%	-	5.50	51.83	42.67	100.00
1997	km^2	-	4.217	7.205	11.300	22.722
	%	-	18.56	31.71	49.73	100.00
2008	km^2	-	-	4.461	18.261	22.722
	%	-	-	19.63	80.37	100.00
2010	km^2	-	-	-	22.722	22.722
	%	-	-	-	100.00	100.00
2017	km^2	-	-	-	22.722	22.722
	%	-	-	-	100.00	100.00

If, in the first year of observations (1977), an area with exceedances of 5 x MAC (5,417 km^2) is revealed, due to the general tendency of decreasing concentrations of Pb, in the following years surfaces are not so highly polluted. Surfaces with Pb concentrations in groundwater between 3-5 MACs are recorded in 1977-1997 and between 1-3 MACs in 1977 - 2008. After 2008, there are no more surfaces with groundwater polluted with Pb, the concentrations determined being below the MAC value.

Correlation of Pb polluted surfaces - time. The percentage of polluted surfaces, for which the Pb concentration of the groundwater exceeds the MAC, shows over time a polynomial second degree trend (Figure 5). This is statistically distinct significant (R^2 = 0.9257).

If, at the beginning of the observation period, 37.64% of the surveyed area did not present Pb pollution problems, in the following years, the polluted areas decreased to: 57.33% in 1990 and 50.27% in 1997 and respectively in 2008 to reach 19.63%. In 2010 and 2017 there are no more polluted areas.

The rate of reduction of the surfaces for which the groundwater is polluted with Pb, resulting from the shape of the correlation curve is 1.89%/year, corresponding to 430 ha/year.

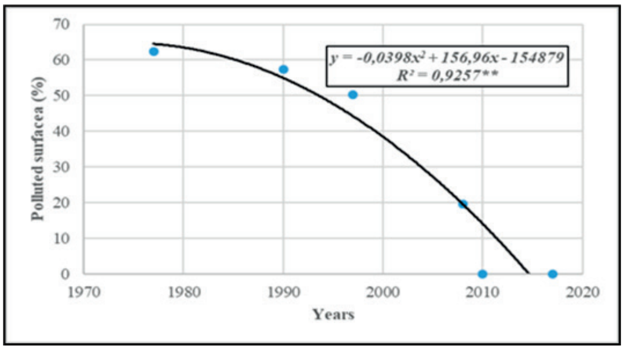

Figure 5. Correlative link between the percentage of polluted surfaces with Pb and time

CONCLUSIONS

The annual average values of SO4 shows the most frequent exceedances of the MAC (250 mg/l) in 7 cases at the P1 drilling in the central part of the analyzed area. The tendency highlighted by the shape of the correlation curve, at the F6 drilling (polynomial of degree III), indicates that from the first year of analysis, the determined concentrations are reduced until 1990, when it is very close to the MAC, after which there is a tendency of growth up to in 2008, then to show a decreasing trend.

Before 1990, the year of separation from the communist period, the area with water contaminated with SO_4 was about 60% (13.934 km^2) of the analyzed area, so that, after restructuring the industrial objectives in the area, it decreased in the last year of observations (2017) to about 10% (2.264 km^2).

The concentrations of Pb in groundwater recorded during the 40-year observation period, at hydrogeological wells in the industrial area of Oradea are between 0.0001 mg/l (P5, P9, F5) and 0.191 mg/l (P1).

The analysis of the average annual values of Pb concentrations in groundwater shows that they exceed MAC: in the first year at 6 drillings; in the second year at 7 drillings and in the third year at 6 drillings.

The exponential correlations established between Pb concentrations at F5 and F6 wells (located in the vicinity of SCF at distances approximately equal to the Oradea-Borș road) and respectively time indicate the annual rate of decrease of the higher Pb concentration at F5 drilling (0.0008 mg/l/year) than at F6 drilling.

The spatial representation of Pb-polluted areas indicates that they are located in the central area of the studied perimeter, near the alleged sources of pollution, SCF and AMP (F6) and on the direction of the underground waters drainage, passing through this area.

At the beginning of the observation period, 1977, the surface polluted with Pb was 62.36% (14.169 km^2), being registered the exceed of 5 x MAC. Over time, the polluted surfaces are reduced, so that in 2010 and 2017 there will be no polluted surfaces.

The quality of groundwater in Oradea's industrial area has improved as a result of the alignment of the Romanian environmental legislation with the EC requirements and the modernization and restructuring of the main industrial enterprises after the year 1990.

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