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SECTION ECOLOGY AND ENVIRONMENTAL PROTECTION

SOIL AND GROUNDWATER CONTAMINATION AROUND THE LOJANE As-Sb MINE, REPUBLIC OF MACEDONIA

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ABSTRACT

The Lojane arsenic-antimony deposit with its former mine facilities, for a longer period, represents source of contamination with heavy and toxic metals into the air, water and soil. Remains of mine waste in Suvi Potok near the Vakcince village, open waste and concentrate storage pond from the Lojane Mine and storage of reagents and toxins near the Tabanovce village represent source of pollution with As, Sb, Hg, Tl, Pb, Mo, Mn etc., which directly threaten surface and ground water in the vicinity of Vaksince, Lojane and Tabanovce as well as soil on a wider area between those three villages. In the groundwaters, the concentrations are: As 16-402 mg l^{-1}, median 33 mg l^{-1}; Sb 0.1-166 mg l^{-1}, median 0.6 mg l^{-1}; Mo 0.3-3.5 mg l^{-1}. For all samples there is a close positive correlation between As and Sb (correlation coefficient 0.9915), but molybdenum have shown strong correlation to As (0.871) and Sb (0.8079) as well.

Twenty-two samples of soil and sediment from locations from the mine dumps exhibit some very high concentrations of As (median 132 mg kg^{-1}) and Sb (median 89 mg kg^{-1}). Concentrations of Mg, Cr and Ni are also high in many of these samples (MgO up to 18.32 wt%, Cr up to 595 mg kg^{-1}, Ni up to 1119 mg kg^{-1}), no doubt illustrating the influence of the serpentinite on the samples. Chromite was noted as being an HF-insoluble phase during the chemical analysis of many of the samples. Statistical analysis performed on results of soil and sediment results have shown that the highest correlation coefficients, as it was expected, occur for As-Sb (0.9924), As-Mo (0.9851), As-Tl (0.9820), As-Cu (0.8535), As-Pb (0.8215), Sb-Mo (0.9943), Sb-Cu (0.8784) and Sb-Pb (0.8301). Beside the statistical parameters, which defined relations between particular elements remains the fact that the highest concentrations in analyzed grounwater and soul samples showed arsenic and antimony as main pollutants and toxic metals in this area.

Keywords: Lojane, pollution, toxic metals, soil, groundwater.

INTRODUCTION

The Lojane deposit is located in the north of Macedonia, close to the border with Kosovo and between the villages of Lojane and Vaksince (Figure 2 and 3). The mine exploited As, Sb and Cr during the period 1923-1979. Sb and As were mined from steeply dipping, NW-SE trending veins, often located at the junction of the andesite and serpentinite [1], [2], [3]. The exploitable veins extend for about 0.5 km and their thicknesses vary between 1-2m. The main ore minerals were the sulphides of As and Sb

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(orpiment, realgar, stibnite), but bravoite ((Fe, Ni, Co)S_2), vaesite (NiS_2), cattierite (CoS_2), pyrite, marcasite, molybdenite and pitchblende (UO_2) also occur in minor quantities. The gangue consists of quartz and minor dolomite. Some oxidation of the primary sulphides has resulted in the formation of secondary oxides and hydroxides. Waste material is estimated at 20 000 t at the mine site [2].

The mined ore used to be transported by rail from the underground workings to the mill and flotation plant and then exported by rail. Considering the beneficiation facilities, remains found on place clearly indicate that flotation process was used. Antimony and arsenic minerals were concentrated by flotation process while generated wastes, known as tailings, were disposed in dry form on the pile created just in front of the plant (Figure 1). The tailings pile covers a surface of app. 17 500 m^2, and contains app. 450 000 tons of tailings. A concentrate storage pond (stone structure with estimated volume of app. 5 000 m^3) build near the plant contains app. 15 000 tons of 50% As concentrate and 3 000 000 t of tailings at the concentration facility [2]. The tailings dam is completely unprotected and its orange colour suggests a high concentration of arsenic sulphides. Concentrate was then taken by small gauge railway across the agricultural area to a location next to the main railway line at Civluk. At this location there are also the remains of the old arsenic smelter (Figure 1).

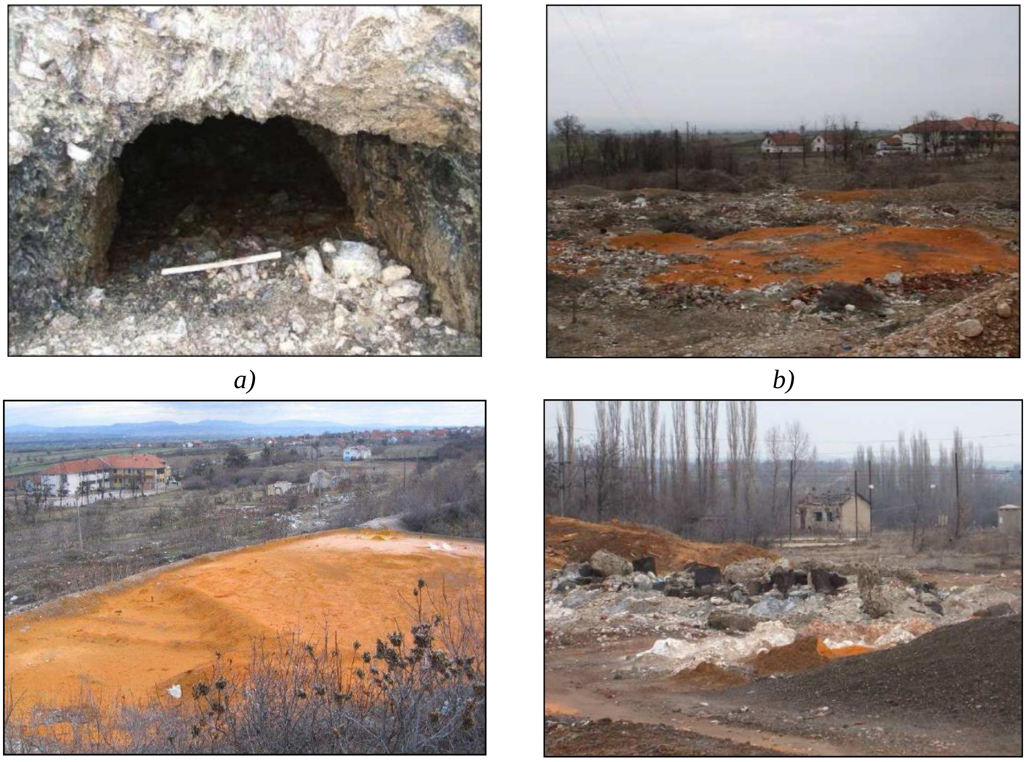
a) *b)* *c)* *d)*

Fig. 1. a) Old Lojane mine adit; b) Arsenic remains near the flotation plant; c) Arsenic concentrate pond near the Lojane village primary school; d) Roasting/smelting facilities remains at the Civluk locality.

The smelter site contains a large pile of arsenic concentrate and is also covered with numerous heaps of black and white powder. These consist of stibnite concentrate and refined ‘white arsenic’ (arsenolite, As_2O_3) and are reputed to be the remains of 2500 wooden barrels of processed material which were abandoned during the recent conflict and left to rot. The facility ruins and storage yard covers surface of 8,800 m^2.Some of the preliminary publications that have studied the issues of the Lojane mine, its production and latter on pollution, can be found in [1], [2], [3], [4], [5] and [6].

GEOLOGICAL SETTINGS

The Lojane deposit area studied consists of a complex of Paleozoic metamorphic rocks and magmatites, a complex of Mesozoic sediments and magmatites, a complex of Tertiary sediments and volcanics as well as Quaternary layers and volcanics (Figure 2).

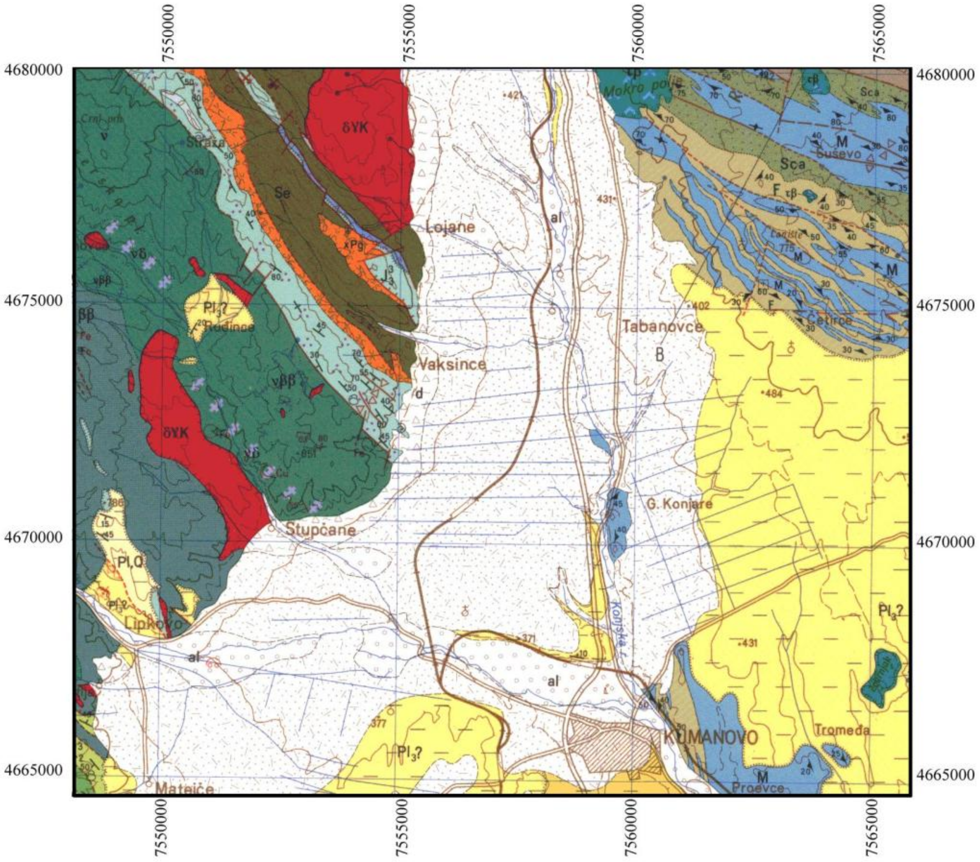

Fig. 2. Geological map of the Lojane area

al-Quaternary alluvium; *d*-Quaternary diluvium; *Pl,Q*-Neogene pebble-carbonaceous limestone; *Pl*$_3$-Neogene sand, clay, sandstone; *xPg*-Paleogene rhyolite; *δγK*-Cretaceous granodiorite; *J*$_3$-Jurassic limestone; *ßß*-Jurassic diabase; *νßß*-Jurassic gabbro-diabase; *ν*-Jurassic gabbro; *Se*-Paleozoic serpentinite; *Sca*-Paleozoic metamorphosed sand and sanstone; *F*-Paleozoic phyllite schist; *M*-Palezoic marble

The *Paleozoic* occupies an important part of the area and is represented by marbles, sericite and phyllitic schist, metamorphic sandstones and serpentinite [2], [3]. Marbles (*M*) that are common in the schists of the Veles series and occur as chert layers and large masses with some regularity. They are white and grey in color, usually made up of large calcite grains with small limonite admixtures. Sericite and phyllitic shists (*F*) are the most widespread of all Paleozoic schists and under the Tertiary sediments they emerge on the surface as erosion windows. They most probably were formed by pelite and psamite metamorphosis. Their mineral composition is simple: quartz, chlorine, muscovite and a little albite and zircon. Metamorphic sandstones (*Sca*) were made up of 65% quartz, 30% muscovite while other component were feldspar and ferruginous material (goethite and hematite), with calcite as cement. Serpentinite (*Se*) is one whole with several ruptures separated in several masses extending to north-west as tectonic interwoven serpentine lenses. Serpentinites are present as dunites and harzburgites while most of their masses were serpentinized. Olivine is the primary component part and chromite secondary. The secondary minerals are serpentinite and magnetite, seldom chlorite and carbonate.

The *Mesozoic complex* of sediments and magmatic rocks is represented by gabbro-diabase, diabase, reef limestone and granodiorite. A gabbro-diabase (*ßß*) zone is a zone of mixture of diabase veins in gabbro and vice versa, gabbro loads in diabases. Diabases (*ßß*) are weathered, broken and cracked. They have dark green, fine-grained structure and massive texture and usually were made of plagioclase grains with inter space filled with pyroxene and plagioclase crystals. Magnetite and limonite occur as accessory minerals. Facies of reef limestones (J_3) are grey-white, cracked and filled with calcite veins while the texture of the rock is fine-grained, of carbonate composition and pselite to psephite structure with some clayey substance. Granodiorites (*K*) occur at two places as large masses, near the villages of Lojane and Slupcane, as medium sized, grey to greenish color conditioned by the feldspar color. The major minerals are plagioclase, orthoclase and quartz. Orthoclases occur as large fresh grains with fine plagioclases in them.

Paleocene complex consists of effusive rocks such is rhyolite (*xPg*). They occur as intrusions through the gabbro diabase massif. They are of porphyry structure with quartz, orthoclase and plagioclase and the colored representatives are biotite and amphibole. They are light or dark grey depending on the alteration type.

Upper Pliocene is present as sandy-clayey series (Pl_3), which consist of sand, clay, loam, lenses of sandstone, limestone and thick gravel layers. The sands are grey-white to yellowish and quite unbound. Clays alternate with sands irregularly occurring as large and longer but thin lenses without defined stratigraphic level. The gravel series (*Pl,Q*) overlies the sand series in most of the terrain and the limestones that occupy the top most part of the sand series. Deluvial layers (*d*) are transition zones in the mountain massifs in other Quaternary layers (proluvial, alluvial etc.). The base is of unworked pieces of various materials but they are filled with clay humus material. Present day alluvial layers have formed at the divide of all major rivers. They are present as gravels. The allusions of gravel material are rare occurring in the lower river courses.

Since the area of interest was for long time in history mining area the spatial distribution of all metal and non-metal occurrences in the area are given. Ore occurrences in the area consist of antimony and arsenic deposits [1], [2] and [3]. Antimony has been found in two localities-Nikustak and Lojane. At Nikustak antimony occurs in Paleozoic schists as mono-mineralization along the vein type. In the Lojane mine both antimony and arsenic have been found in the serpentinite which is in contact with rhyolites. Ore bodies are complex ore veins. Major ore minerals are antimony and realgar of 6.5% As and 4% Sb.

METHODOLOGY

Samples were collected from the Lojane area locations outlined below, with a greater emphasis placed on soil sampes. Sample locations are given on a map (Figure 3). Samples were taken progressively downstream from the mining site and, at some locations, in receiving waters. Sampling density varied from a few metres to several kilometres, depending on the situation; in general, sampling density was lower further downstream. Water was collected in polythene syringes, passed through a 0.45μm filter and transferred into polythene bottles. Conductivity and pH were measured in the field for all water samples and then water was acidified with 0.4 ml of 50% nitric acid. Samples were stored in a cool, dark place until they reached laboratory for analysis. Soil and sediment samples were also collected at several locations. These were dried, ground and then dissolved using a hot HF-$HClO_4$ digestion. Solutions were analysed by ICP-AES or ICP-MS, depending on concentrations. A large number of analytes were determined but only those that are likely to be mining-related and environmentally significant are presented and discussed here.

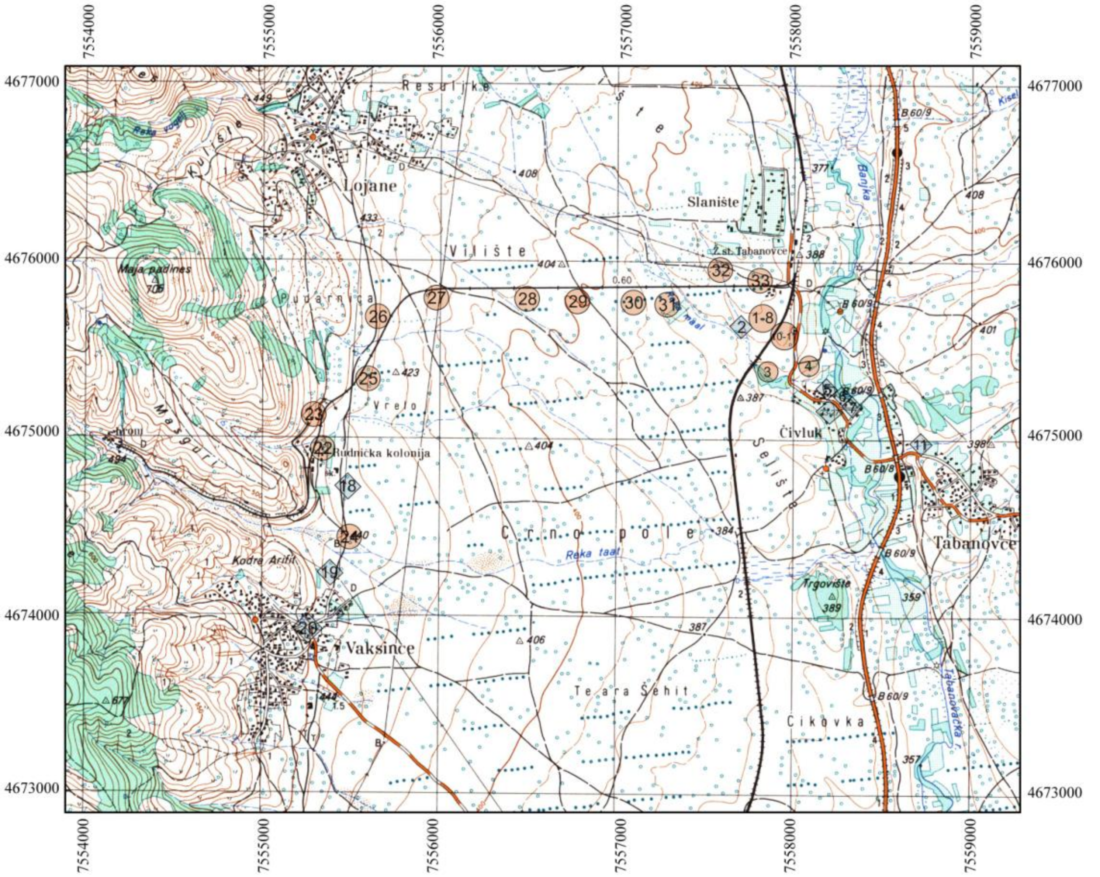

Fig. 3. Geological map of the Lojane area

◆ - water samples; ○ - soil and sediment samples

The accuracy of the soil and sediment results was assessed by routinely analysing standard soil samples. There was some concern that As and Sb might be lost by volatilization during the solid dissolution procedure, so selected samples were also analysed by X-ray fluorescence. It was found that some As does get lost, but this only becomes significant at levels greater than about 100 mg kg^{-1} and then the amount lost equates to about 10%.

The concentrations of As and Sb in the soils, sediments and waters were compared to various national screening and reference guidelines to assess their significance [7], [8], [9] and [10]. There is some variation in the values for these guidelines and the data for Sb are not as comprehensive as those for As. This is partly because the values may be set for local conditions, but also because they have been derived using different ecotoxicological tests or for different applications (e.g. drinking water, irrigation, environmental well-being, intervention).

RESULTS AND DISCUSSIONS

Groundwaters collected from the Lojane area (13 samples) are all alkaline with a pH of 7.6 to 8.3 (Table 1). A distinctive feature of their chemistry is that for most samples Mg cncentrations are higher than those for Ca while high conductivities (mostly >610 μS cm^{-1}) point to a high dissolved solid content. These features are likely due to the interaction with certain underlying, relatively reactive lithologies, particularly serpentinite (containing abundant Mg silicates) as well as dolomite/limestone.

Table 1. Chemical analysis of ground waters from the Lojane area.

Sample	Al (mg l^{-1})	As (μg l^{-1})	Ca (mg l^{-1})	Fe (mg l^{-1})	K (mg l^{-1})	Mg (mg l^{-1})	Mn (mg l^{-1})	Mo (μg l^{-1})	Na (mg l^{-1})	Sb (μg l^{-1})	pH	C (μS/cm)
2g	0.1	20.5	47.1	<0.1	18.8	64.6	<0.1	0.1	7.3	0.8	7.7	610
5g	<0.1	19.9	44.8	<0.1	1.5	64.1	<0.1	0.3	7.8	0.1	7.6	630
6g	0.1	40.5	71.2	<0.1	6.0	114.0	0.3	1.2	16.5	0.5	7.7	1100
7g	0.1	26.1	68.1	<0.1	1.2	101.6	<0.1	0.6	10.8	2.0	7.7	940
11g	0.1	29.8	63.6	<0.1	18.6	81.8	<0.1	1.5	13.0	0.6	7.8	860
18g	0.1	70.4	29.1	<0.1	7.4	82.4	<0.1	2.2	5.4	8.0	8.3	705
19g	<0.1	402	25.4	<0.1	4.5	110.4	<0.1	3.5	4.0	166.3	8.2	875
20g	0.1	54.6	71.6	<0.1	49.0	78.4	<0.1	0.7	19.0	5.7	8.0	1050
23g	<0.1	60.4	64.7	<0.1	1.6	100.6	0.3	1.3	10.6	0.8	7.6	958
24g	<0.1	26.1	49.0	<0.1	1.0	67.7	0.1	0.8	5.8	0.5	7.8	668
25g	<0.1	35.9	46.0	<0.1	1.6	62.4	0.1	0.6	6.8	0.6	7.8	621
26g	<0.1	22.7	44.7	<0.1	1.6	62.9	<0.1	0.6	6.5	0.6	7.8	683
27g	<0.1	16.4	51.4	<0.1	3.2	68.2	<0.1	0.4	6.3	0.4	7.7	682
min	0.1	16.4	25.4	0.1	1	62.4	0.1	0.1	4	0.1	7.6	610
max	0.1	402	71.6	0.1	49	114	0.3	3.5	19	166.3	8.3	1100
median	0.1	29.8	49	0.1	3.2	78.4	0.3	0.7	7.3	0.6	7.8	705
average	0.1	63.485	52.054	0.1	8.923	81.469	0.238	1.062	9.215	14.377	7.823	798.615

In the groundwaters, the concentrations are: As 16-402 mg kg^{-1}, median 33 mg kg^{-1}; Sb 0.1-166 mg kg^{-1}, median 0.6 mg kg^{-1}; Mo 0.3-3.5 mg kg^{-1}. For all samples there is a close positive correlation between As and Sb (correlation coefficient 0.9915), but molybdenum have shown strong correlation to As (0.871) and Sb (0.8079) as well. There appears to be no major variation in concentration of either As or Sb with pH (Table 1). Most of the waters have As concentrations which exceed the 10 μg l^{-1} value accepted for safe drinking water (UK standard) and 5μg l^{-1} MDKvalue (Macedonian standard). In contrast, most Sb concentrations are much lower than 10 μg l^{-1} and appear to be within the acceptable limits for this element. Waters discharging from adits or in close proximity to the tailings and smelting sites have higher concentrations of the elements As, Sb and Mo. Some waters sampled from wells (and number 19g from Vaksince in particular) have contents of As and Sb which are significantly above the drinking water limits set by the various regulatory agencies. However, we would like to stress out that beside certain influence of former anthropogenic mine activities increased concentrations of As, also, are results of high natural background of this element, and all wells drilled in serpentine are naturally contaminated. Even in the wells from the zones considered to be none affected with former mining activities, similar concentrations are detected.

Twenty-two samples of soil and sediment from locations separate from the mine dumps exhibit some very high concentrations of As (median 132 mg kg^{-1}) and Sb (median 89 mg kg^{-1}) (Table 2). Location 11 is particularly noteworthy (As > 10 000 mg kg^{-1} and Sb > 7 000 mg kg^{-1}); Mo and Tl values are also particularly high in that sample. Overall, concentrations of As tend to be higher than those for Sb (Table 2). The concentrations of As and Sb across the study area are quite variable and even replicate samples from the same location can have concentrations of both of these elements which vary by at least 50%. Many of the soil and sediment samples have As and Sb contents which exceed proposed environmental quality limits (typically around 20 mg kg^{-1} for both elements), even if a rather conservative threshold value of 50 mg kg^{-1} for both Sb and As is adopted, it is clear that many samples have contents of these elements which are above the 'intervention' values and thus classed as 'contaminated' and requiring remediation. This is very similar to findings of [5] where in tailings As ranged 8400-9900 mg kg^{-1} and Sb 6600-6800 mg kg^{-1}, while in As-concentrate remains near the former flotation plant were determined concentrations of 505000-511000 mg kg^{-1} As and 63500-68600 mg kg^{-1} Sb and in topsoil 230 mg kg^{-1} As and 260 mg kg^{-1} Sb. Also, this data conforms with data found in [11], [12] and [13].

Table 2. Chemical analyses of soils and sediments from the Lojane area.

Sample	**Fe_2O_3 (wt %)**	**MgO (wt %)**	**CaO (wt %)**	**MnO (wt %)**	**As (mg kg^{-1})**	**Ba (mg kg^{-1})**	**Cr (mg kg^{-1})**	**Cu (mg kg^{-1})**	**Mo (mg kg^{-1})**	**Ni (mg kg^{-1})**	**Pb (mg kg^{-1})**	**Sb (mg kg^{-1})**	**Tl (mg kg^{-1})**	**Zn (mg kg^{-1})**
1	3.46	1.71	4.43	0.05	414	547	82	16	0.6	82	25	201	1.5	42
2	5.67	1.91	1.33	0.12	1635	509	140	28	1.3	128	39	625	2.2	73
3	5.47	1.80	1.21	0.12	298	525	135	25	0.3	105	38	60	0.7	67
4	5.20	1.59	1.09	0.11	115	501	130	26	0.3	100	37	23	0.5	64
5	4.95	1.44	0.94	0.10	95	515	135	24	0.1	99	33	11	0.4	56
6	3.44	5.18	1.03	0.12	147	491	109	30	0.1	272	26	12	0.2	53
7	5.32	8.50	2.07	0.11	47	405	330	37	0.1	428	37	7	0.2	62
8	3.81	4.86	1.32	0.09	31	359	301	30	0.1	280	39	6	0.3	52
10	5.10	2.19	1.46	0.10	937	444	126	37	1.3	124	47	596	2.2	76
11	10.32	1.75	1.47	0.08	10838	391	162	105	24.0	144	134	7593	32.4	110
22	6.46	18.32	8.49	0.09	1009	196	428	25	0.8	1119	101	605	5.5	59
23	4.65	7.80	19.67	0.10	1611	234	231	34	0.9	485	32	749	2.5	72
24	6.47	16.57	6.20	0.11	81	252	320	31	0.2	895	39	35	0.1	89
25	6.03	3.76	2.57	0.24	117	413	464	56	0.6	379	51	104	3.3	80
26	4.94	3.30	13.90	0.14	94	305	269	39	0.6	403	36	107	1.5	72
27	6.49	3.84	2.27	0.26	333	467	595	41	1.5	776	43	837	3.3	72
28	6.59	3.01	1.60	0.16	42	434	381	38	0.5	582	35	35	0.9	70
29	5.45	6.80	2.65	0.14	74	395	345	30	0.4	575	27	74	0.7	58
30	5.53	3.72	2.43	0.11	51	403	345	35	0.4	364	34	31	0.9	61
31	5.79	3.61	1.95	0.10	48	477	398	29	0.5	384	33	21	0.9	58
32	4.10	3.40	2.56	0.06	161	441	387	19	0.6	202	33	128	1.1	50
33	5.07	2.80	1.73	0.10	216	531	243	28	0.8	203	41	174	1.2	66
min	3.44	1.44	0.94	0.05	31	196	82	16	0.1	82	25	6	0.1	42
max	10.32	18.32	19.67	0.26	10838	547	595	105	24	1119	134	7593	32.4	110
median	5.385	3.505	2.01	0.11	132	437.5	285	30	0.55	322	37	89	1	65
average	5.469	4.903	3.744	0.119	836.091	419.773	275.273	34.682	1.636	369.5	43.636	547	2.841	66.455

Concentrations of Mg, Cr and Ni are also high in many of these samples (MgO up to 18.32 wt%, Cr up to 595 mg kg^{-1}, Ni up to 1119 mg kg^{-1}), no doubt illustrating the influence of the serpentinite on the samples. Chromite was noted as being an HF-insoluble phase during the chemical analysis of many of the samples. Statistical analysis performed on results of soil and sediment results have shown that the highest correlation coefficients, as it was expected, occur for As-Sb (0.9924), As-Mo (0.9851), As-Tl (0.9820), As-Cu (0.8535), As-Pb (0.8215), Sb-Mo (0.9943), Sb-Cu (0.8784) and Sb-Pb (0.8301).

CONCLUSION

As we could conclude from ours latest study the vicinity of the Lojane mine area is the main potential threat to the environment, because of the size of the former operation, the close proximity to human activity and the nature of the terrain. Namely, the main problem is from As and to a lesser extent Sb, due to former element’s greater volume, mobility and (probable) enhanced toxicity. There are also some elevated levels of Tl and Mo in these samples. The prevailing portion of the samples at Lojane Mine area can easily be classified as hazardous due to high concentrations of contaminants identified (As, Sb, Ni, and Tl potentially) as well as due to their high leachability (except some waste rocks; [5], [6], [13]). However, the results from this study suggest that Tl and Mo concentrations in water and soils/sediments away from the immediate mine sites are probably not high enough to pose a significant threat. There are also some high levels of Cr in the Lojane soils and sediments but these arise from presence of chromite and they should not present any problem because of this mineral’s stability during weathering.

At the end, based on the data collected within the frame of this and some previous studies we may point out that high environmental and health risks were posed by the mining/ore processing wastes insecurely dumped in both the contamination source zones such are Lojane Mine tailings dump and the area of former roasting facility near the Tabanovce/Civluk locality. This material is exposed to the weather for continued physical transport via rivers and wind-blown dust, and chemical weathering and transport in solution, which increases its environmental impact threat.

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