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INVESTIGATION OF HEAVY METAL MOBILIZATION AND ITS ENVIRONMENTAL IMPACT
ON THE MINE SITE OF RUDABÁNYA

PhD thesis

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I. Research work

The main goal of my research work is to find answer to the question how the tailings of Rudabánya influence the water quality of surface and subsurface water bodies. I created an investigation sequence where firstly I determined the amount of different heavy metals in different fractions by sequential extraction. The result of this investigation proved that selected tailings have notable mobile heavy metal content so in the next column test I determined the amount of heavy metals which could be mobilized by distilled water. This experiment proved that some heavy metals could be washed out from the tailings and they could be spread by the percolating water. To know this process I created another column test where I determined the transport parameters of investigated heavy metals in the tailing material of Rudabánya. I had no possibility to investigate the heavy metal content of groundwater but I have operated a one-and-a-half-year long monitoring system in the Lake Rudabánya to know the chemical composition changes of surface water. The graphical abstract of research work can be seen on Figure 1..

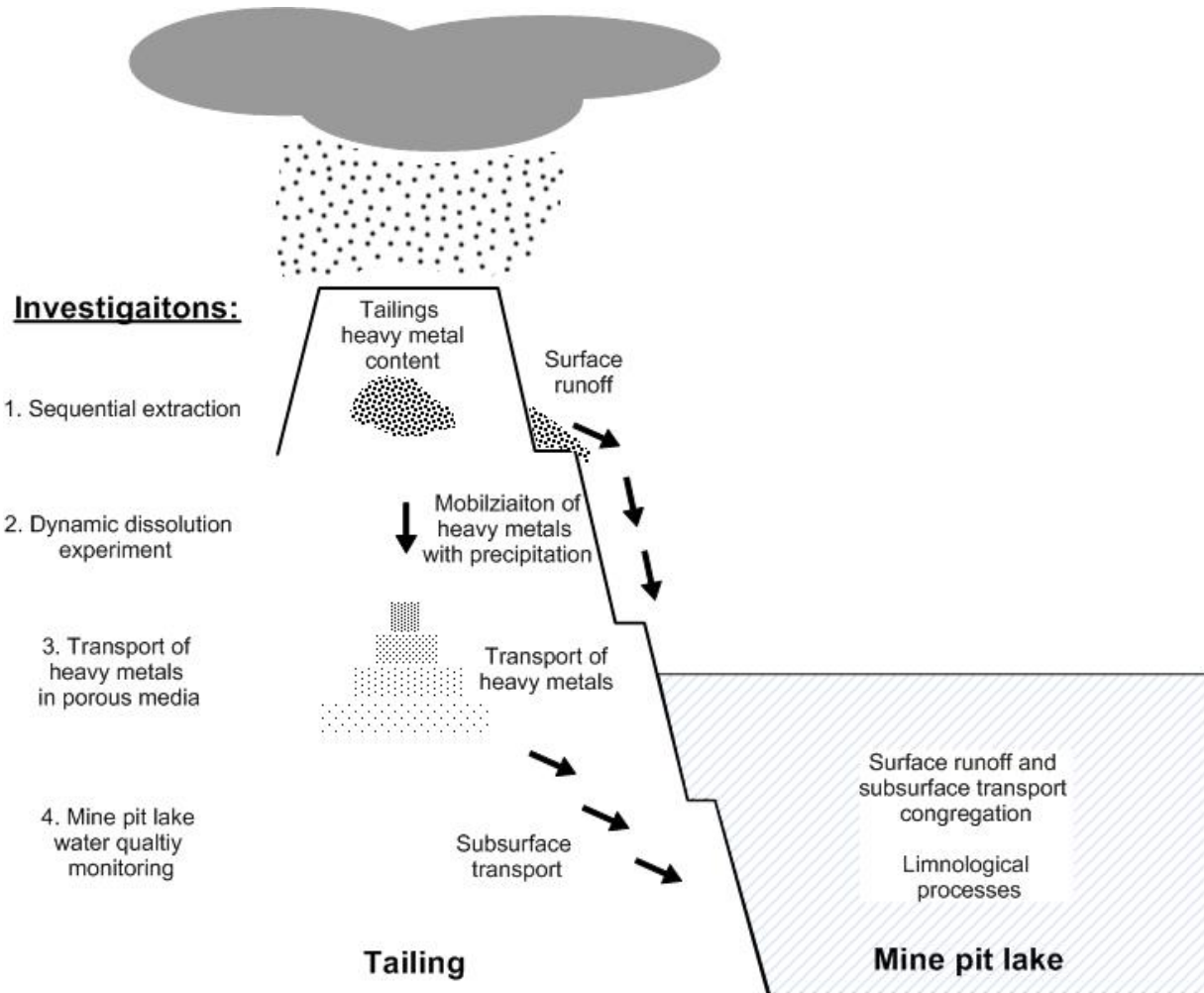


Figure 1. Graphical abstract of the research work

II. Applied methods

Sequential extraction

During investigation of contamination of a soil or a sediment sample the question is the concentration of heavy metals in them. But the total concentration of a heavy metal is not information because it could have different forms in the sample at the same time. The name of this phenomenon is speciation which means the distribution of a heavy metal between its physical chemical forms. The tools which could be used to separate the heavy metals according to their physical chemical properties into fractions are sequential extractions.

In my research work I applied two sequential extraction methods. They were the Hungarian standard, MSZ 21470-50:2006 (MSZ-21470-50, 2006) and the modified Tessier sequence (Tessier, et al., 1979). The fractions of MSZ 21470-50:2006 were water soluble, Lakanen-Erviö, nitric acid- hydrogen peroxide and aqua regia fraction, respectively, while the fractions of modified Tessier sequence were water soluble, exchangeable, bound to carbonates, bound to organics and sulphide fractions, respectively.

The element content of extracts were analyzed by a UniCam 929 AA spectrophotometer.

Dissolution experiments

Distilled water were percolated through soil columns prepared from the tailing material of Rudabánya. This experiment were performed in case of saturated and unsaturated conditions, respectively. The element concentration of effluent fluid were analyzed by the mentioned UniCam 929 AAS and the results were evaluated by ANOVA.

Determination of transport parameters

The column experiments are often used tools to investigate transport processes of organic and inorganic components (Xiong, et al., 2006) (Chotpantarat, et al., 2011) (Colombani, et al., 2015) (Nguyen, et al., 2015) (Masipan, et al., 2016). The two methods of these experiments are breakthrough curve experiments (BTC) and impulse tests (IT). In case of BTC a solution with constant concentration flows through the column, in case of IT a known amount of substance spreads through the column. The process of BTC experiment could be described by the Equation 1 (Van Genuchten function) while the process of IT could be defined by the Equation 2 (Gauss function).

$$c(x,t) = \frac{C_0}{2} \left\{ e^{-\left[\frac{\left(v - v \sqrt{1 + \frac{4\mu D}{v^2}} \right) x}{2D} \right]} \operatorname{erfc} \left[\frac{Rx - v \sqrt{1 + \frac{4\mu D}{v^2}} t}{2\sqrt{DRt}} \right] + e^{-\left[\frac{\left(v + v \sqrt{1 + \frac{4\mu D}{v^2}} \right) x}{2D} \right]} \operatorname{erfc} \left[\frac{Rx + v \sqrt{1 + \frac{4\mu D}{v^2}} t}{2\sqrt{DRt}} \right] \right\} \quad (1)$$

$$c(x,t) = \frac{M}{2An_0R\sqrt{\frac{\pi\alpha_L vt}{R}}} e^{-\left(\frac{x - \frac{vt}{R}}{\frac{4\alpha_L vt}{R}} \right)^2} e^{-\mu t} \quad (2)$$

where c is concentration of the effluent fluid, C_0 is initial concentration, M is total amount of substance, t is time, D is dispersion constant, R is retardation factor, α_L is longitudinal dispersion, A is surface of column cross-section, n_0 is porosity, x is distance, v is mean velocity of flow and μ is decay constant or irreversible sorption constant.

The derived transport parameters from these column experiments are retardation factor, dispersion constant and irreversible sorption constant.

Some parameter above could be easily measured but some of them could be determined on indirect way. To determine the transport parameters I used the version 8.6.0 of OriginPro of OriginLab Corporation. One tool of this program is to fit know mathematical formulas on measured data. After reaching of the best fitting the non-measurable parameters could be derived.

Whereas the transport parameters are not measured data to know parameter sensitivity of transport equations on transport parameters is non-negligible. In the literature there are several parameter sensitivity investigation method, in my analysis I used the partial derivation method. According to Atherton et al. (1975) if the relation between the dependent and independent variable can be described by an explicit function the partial derivation could be easily performed (Atherton, et al., 1975). In this case the sensitivity coefficient is φ_i :

$$\varphi_i = \frac{\partial}{\partial P_i} F \cdot \left(\frac{P_i}{F} \right) \quad (3)$$

where P_i is one parameter of F function (Hamby, 1994).

Water quality monitoring

In the Lake Rudabánya a one-and-a-half-year long water quality monitoring program were performed where water samples were collected in nine sampling point from different depths. The element concentration were analyzed by the previously mentioned UniCam 929 AA spectrophotometer.

III. Results, thesis

Results of sequential extractions

The results of sequential extractions are summarized on graphs like Figure.2. The bioavailable element content of a sample could be determined by the Lakanen and Erviö solution (Lakanen & Erviö, 1971). This fraction is almost equivalent with the bound to carbonate fraction in the Tessier sequence. According to Kersten, Schintu and Fernández-Ondoño (Kersten, 2002) (Schintu, et al., 2016) (Fernández-Ondoño, et al., 2017) the real bioavailable element content of a sample could be found in the water soluble and exchangeable fraction because the bioavailability of bound to carbonate fraction is depended on the physical and chemical properties of the soil (Kersten, 2002). The sulphides which presence is proved by the sequential extraction are oxidized on the air. The well-known result of this oxidation is the phenomenon of AMD (acid mine drainage) which means serious environmental problems in other mine sites. In Rudabánya the huge amount of carbonate rocks buffers the acid from sulphide decay and they bound the heavy metals as carbonates. So in the geochemical environment of Rudabánya is hard to imagine an environmental impact which could mobilize the heavy metals from the bound to carbonate fraction.

Thesis 1.

With sequential extraction I proved there is easily mobilizable element content of tailings of Rudabánya which are found in both water soluble and exchangeable fraction of them. Even so with sequence of experiments I found these heavy metals cannot experienced in the surface and subsurface water bodies because of carbonate rich geochemical environment of Rudabánya.

Result of dissolution experiments

The results of ANOVA based on concentration of element in the effluent fluid are found in the Table 1..

In this case the water saturation was the main difference between column experiments. The experienced differences in the dissolved ion concentration could be caused by the different redox conditions. Hay et al. (Hay, et al., 2016) performed the same experiment where they investigated the concentration change of selenium and sulphate under saturated and unsaturated percolation conditions. They found lower selenium and sulphate concentration during saturated flow because of more reducible condition as against unsaturated flow. In my experiment I found the same the dissolved concentration of zinc, manganese and sulphate were higher under unsaturated flow

conditions. One part of the dissolved sulphate is originated from the dissolution of secondary sulphate minerals (Dold, 1999) while the other part of it could be originated from the oxidization process of sulphide which could be more intensive about the continuous supply of oxygen from the pores. (Hay, et al., 2016).

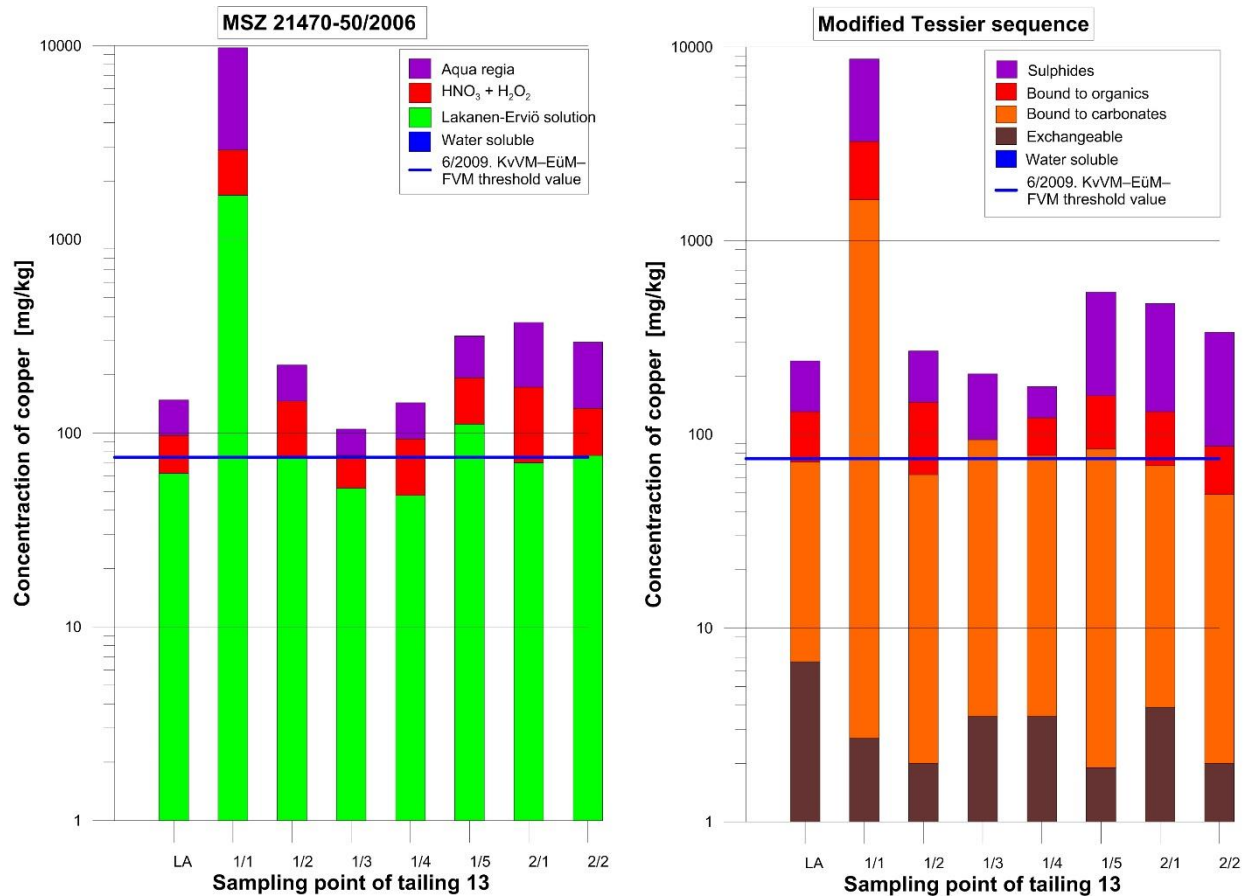


Figure 2. Distribution of copper in different fractions in the samples of tailing 13

Thesis 2.

In column experiments I have found significant differences between dissolved element concentration of effluent fluids percolated through the column under saturated and unsaturated flow conditions. According to this experiment the experienced concentration differences of calcium, magnesium, manganese, zinc and sulphate in effluent fluids are caused by the different way of seeping. This means there is significantly higher dissolved concentration of elements in the unsaturated zone of tailings of Rudabánya than in their saturated zone.

Table 1. The calculated F ratios and the different F values of different error percentage

	F ratio	F 5%	F 1%	F 0,1%
pH	1.2	7.7	21.2	74.1
eH [mV]	1.6	7.7	21.2	74.1
TDS [mg/dm³]	98.7	7.7	21.2	74.1
Ca [mg/dm³]	68.1	7.7	21.2	74.1
Mg [mg/dm³]	80.4	7.7	21.2	74.1
Na [mg/dm³]	193.6	7.7	21.2	74.1
K [mg/dm³]	0.0	7.7	21.2	74.1
Fe [mg/dm³]	0.8	7.7	21.2	74.1
Mn [mg/dm³]	17.7	7.7	21.2	74.1
Cu [mg/dm³]	0.7	7.7	21.2	74.1
Zn [mg/dm³]	41.0	7.7	21.2	74.1
HCO₃⁻ [mg/dm³]	0.7	7.7	21.2	74.1
Cl⁻ [mg/dm³]	2.1	7.7	21.2	74.1
SO₄²⁻ [mg/dm³]	67.8	7.7	21.2	74.1

Results of transport parameter determination

According to breakthrough curve experiment and impulse test there is irreversible sorption in the tailing material of Rudabánya (Figure 3.). This process is more observable in case of iron and copper but in case of manganese and zinc is needed to perform detailed investigation. The mean velocity of seepage was high in the experiments, the seeping velocity generally is slower in the tailings so the irreversible sorption could be more significant on the mine site. To compare the saturated flow labor experiments and the field conditions the saturated flow could be experienced in the deeper zone of tailings above that the flow is happened in unsaturated way which means significantly slower transport of heavy metals.

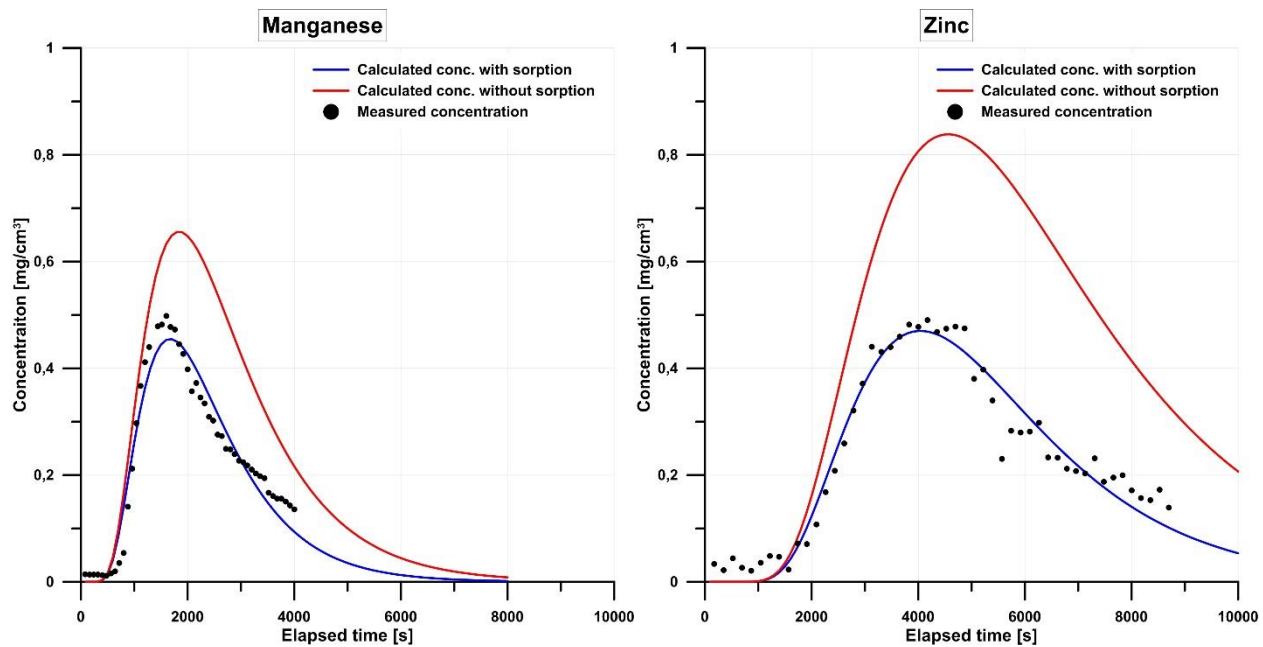


Figure 3. The measured and calculated concentration of manganese and zinc with and without sorption

Thesis 3.

According to breakthrough curve experiments and impulse tests I found there is irreversible sorption or precipitation of iron, manganese, zinc, and copper in the tailing material of Rudabánya during reacting with heavy metal solutions.

During parameter sensitivity analysis I found the Van Genuchten equation has no sensitivity on changes of irreversible sorption (Figure 4.). The highest calculated sensitivity coefficient is 0.09 at 9000 second. This means the changes of irreversible sorption with 1 % causes only 0.09 % changes in the value of calculated concentration. During curve fitting this phenomenon can cause problems because the value of irreversible sorption can change in huge interval which results the same good correlation coefficient of fitting. Contrary, the impulse test has better parameter sensitivity on irreversible sorption which can ensure more accurate results. But it has to take into account the higher parameter sensitivity means higher error sensitivity. In case of applied method the fitting happens on more or less 120 measured points, so the derived parameters calculated from many data point so the positive, negative errors of each measured data eliminate their own effect in the final result.

Thesis 4.

According to parameter sensitivity analysis I proved that Van Genuchten function is less sensitive on changes of irreversible sorption as against Gauss function so during determination of transport parameters in labor experiments the impulse test ensures more accurate results.

Results of water quality monitoring

Physical chemical processes of the lake according to a summer measurement

The physical chemical processes of the lake are shown on Figure 5.. It can be seen the water quality changes of lake could be followed across the general environmental condition indicators.

Changes of environmental condition indicators

The driving force of chemical processes of lake is the density difference caused by temperature induced stratification. This stratification has direct effect on oxygen content of layers. The changes of oxygen with depth is very complex. The literature called this type of oxygen profile (Figure 6.) as clinograde because the concentration of oxygen becomes zero below a depth and it called as positive heterograde because the highest oxygen concentration can be found in the metalimnion (Wetzel, 2001). The reason of latter phenomenon is oxygen loss from the epilimnion about the higher water temperature and oxygen loss in hypolimnion about the oxidation of organic matters by algae. The oxygen saturation above 100 % indicates the oxygen production of aglae in the metalimnion (Wetzel, 2001). The oxygen concentration starts to decrease between depth of 13 and 14 m and at depth of 16 m it is almost zero. The redox potential follow the oxygen profile and it starts to decrease at depth of 18 m.

The other environmental condition indicator which is influenced by photosynthesis is pH. The lakes could be divided into trophogenic and tropholitic zones. The trophogenic zone where the photosynthetic production happens and the tropholitic zone is the zone of decomposition of organic matter(Wetzel, 2001). The

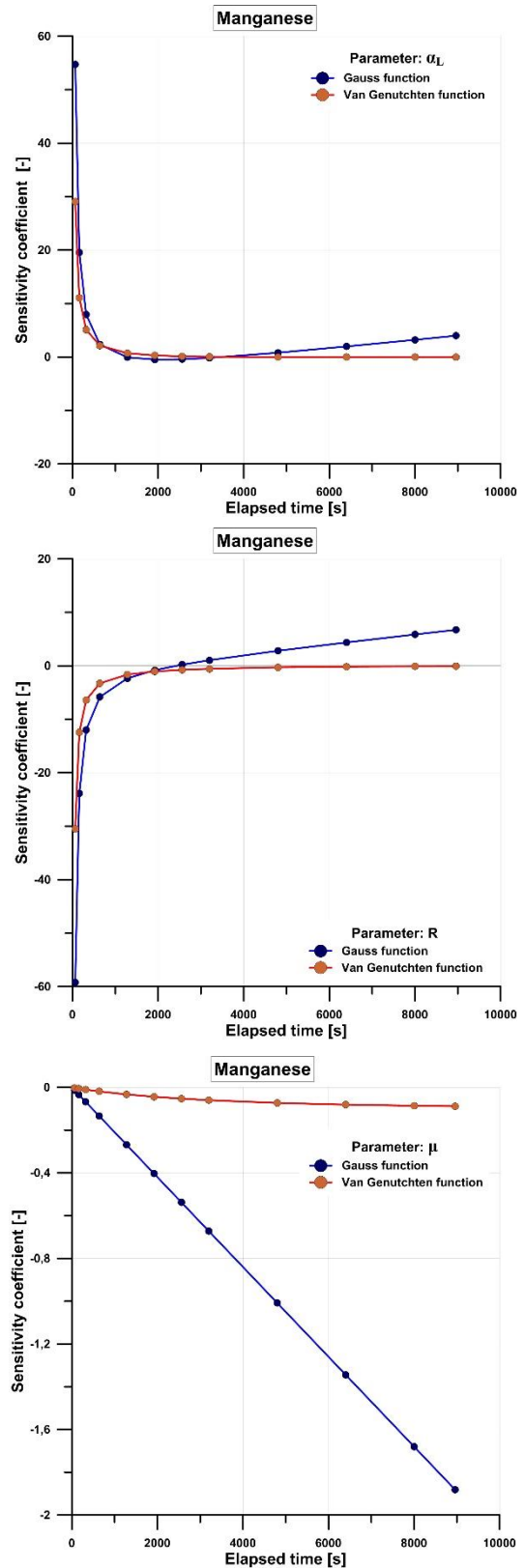


Figure 4. Parameter sensitivity of α_L , R és μ

photosynthesis decreases the amount of dissolved CO_2 and increase the pH contrary the decomposition of organic matter increase the amount of CO_2 and decrease the pH (Wetzel, 2001). Between 0 and 12 m the photosynthesis continuously consumes the CO_2 from water the pH only decrease in small steps (Figure 6.). On the border of hypolimnion and metalimnion the concentration of oxygen starts to decrease because there is no supply from the

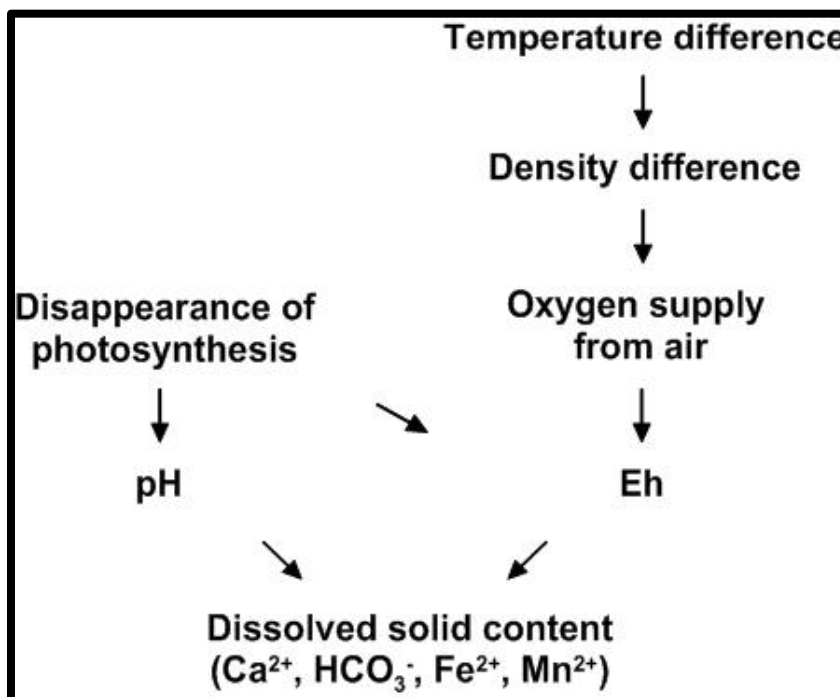


Figure 5. Factors of water chemistry

air and there is no enough light for photosynthesis (Wetzel, 2001). Between depth of 13 m and 16 m all amount of oxygen is consumed the heterotrophic decomposition of organic matters takes place and the produced CO_2 decreases the pH. The highest gradient in pH change can be experienced between depth of 13 and 16 m (Figure 6.). The changes of pH and redox conditions cause changes in the amount of total dissolved solids. The immediate increasing of electric conductivity can be experienced in the same depth between 13 and 16 m (Figure 6.). Decreasing of redox potential also increases the amount of total dissolved solids but the affected elements are micro elements like manganese or iron and their amount is not so significant as against the macro elements like calcium and bicarbonate.

Changes of water composition

The calcium and bicarbonate are dynamic ions and their concentration is affected by metabolism. In hard water lakes like Lake Rudabánya the photosynthesis decreases the amount of CO_2 which causes the precipitation of calcite in the trophogenic zone. This mineral sinks into the more acidic tropholitic zone and one part of it redissolves (Wetzel, 2001). On the graph of Ca^{2+} and HCO_3^- this process could be seen (Figure 7, Figure 8).

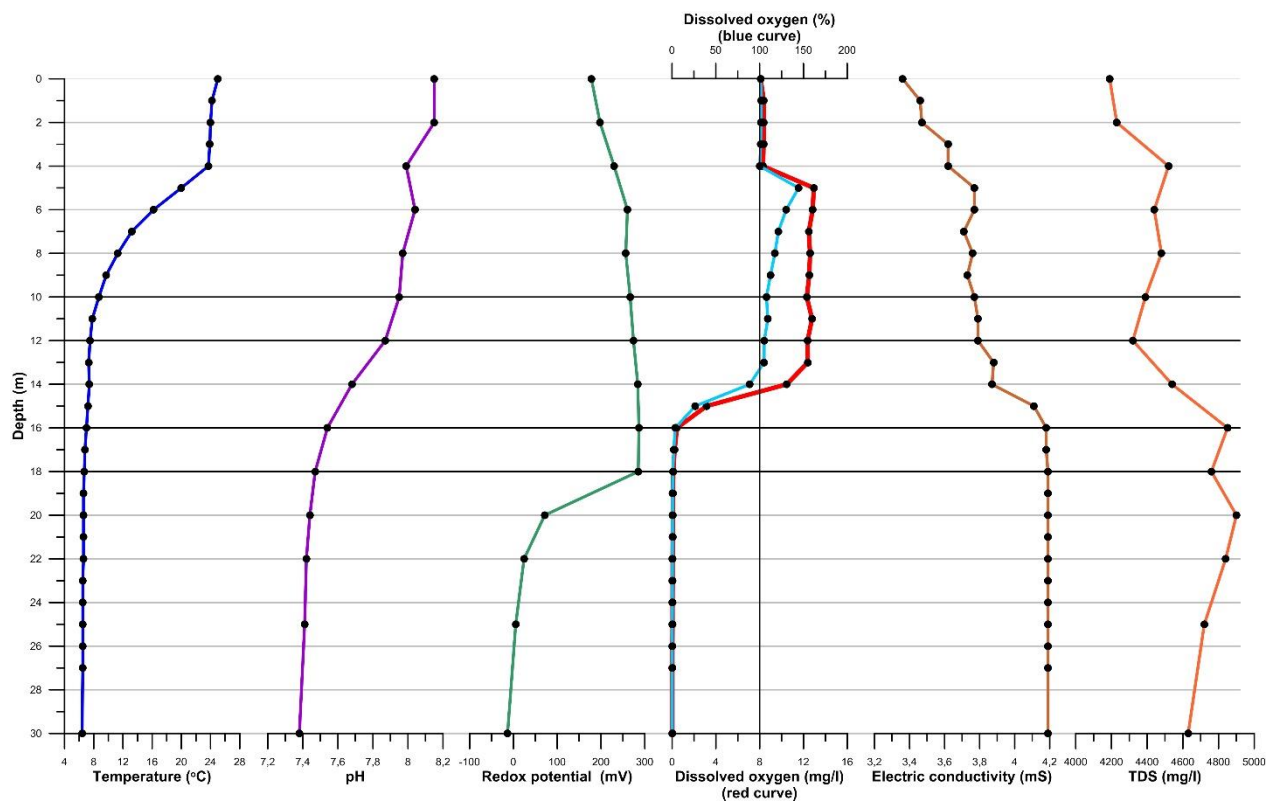


Figure 6. General environmental condition indicators in the sampling point 8 during summer

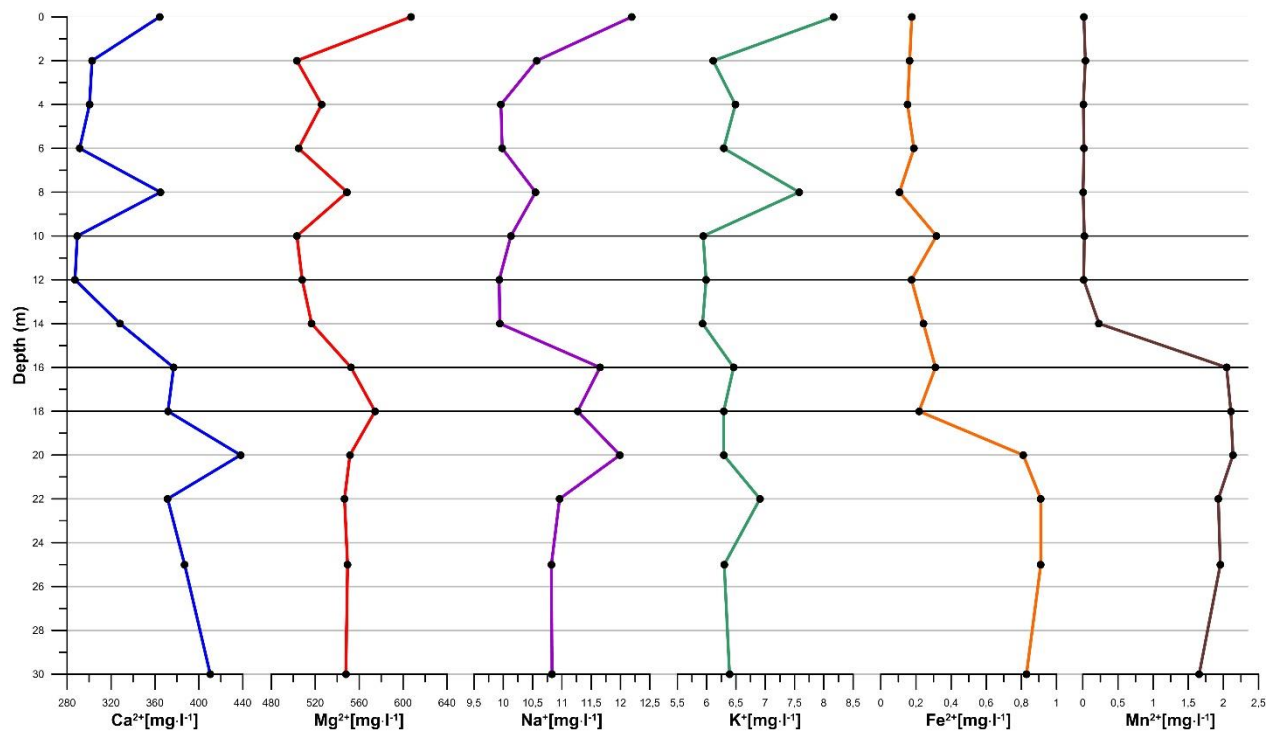


Figure 7. Cation concentration in the sampling point 8 during summer

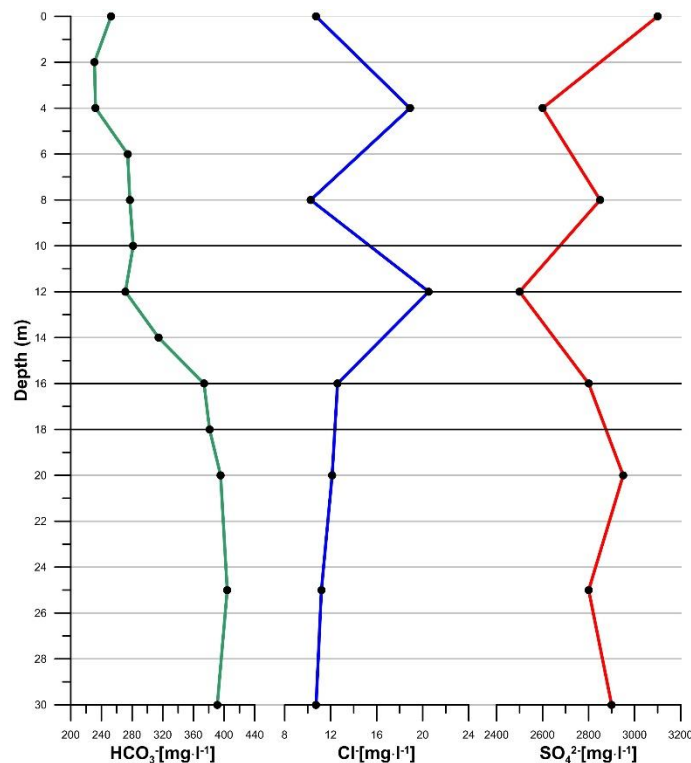


Figure 8. Anion concentration in the sampling point 8 during summer

Seasonal change of water composition

The seasonal changes of water quality can be seen on 1. Figure 9. and 2. Figure 10.. In the sampling point of 7 during summer the pH decreases from the top to the bottom. During autumn this decreasing tendency disappears the pH starts to become homogeneous across the depth-finding profile. In this period the cooling water starts a mixing mechanism which destroys the summer stratification of the lake. During winter there is an inverse layering in the lake according to the literature but I cannot measure about ice covering. From March the stratification starts to form again (1. Figure 9.). In the sampling point 8 this process can also be seen but about the higher depth lower pH values can be experienced at the bottom of the lake.

The dissolved manganese concentration depends on the pH. There is no dissolved manganese in the lake to the depth of 15 m, it can be seen in case of the sampling point 7 because the pH value does not decrease below the value of 7.5 any period of the year (2. Figure 10.). But in case of sampling point 8 the manganese can be found in dissolved form below depth of 15 m from late spring to early autumn (2. Figure 10.). In the mixing periods of lake the manganese can be found in the precipitated form of rhodochrosite.

Thesis 5.

It is proved that the monthly monitoring of pH and manganese in the deepest sampling point of the Lake Rudabánya is enough to deduce to the yearly mixing behavior of lake. According to the yearly pH and manganese cross-sections it can be established the Lake Rudabánya is a holomictic lake and from the aspect of mixing frequency is dimictic lake.

Thesis 6.

From the aspect of stratification the Lake Rudabánya works as a natural lake not like a typical mine pit lake. The main evidence of this statement there is not monimolimnion in the deepest point of the lake.

Applicability of results

Most of my results are site specific related to the mine site of Rudabánya. In my research work I evaluated the heavy metal content of tailings from the aspect of environmental protection. According to my results in the geochemical environment of Rudabánya the transport of heavy metals is strongly inhibited so the heavy metal contamination of surface and subsurface water bodies is negligible.

In the dissolution experiments I proved there is significant difference in the dissolved amount elements during saturated and unsaturated flow conditions. I think this is an information to who would likes to investigate dissolution from soil columns in laboratory.

In case of equation describing breakthrough curve experiment I proved it is important to apply the correct form of 1D transport equation because elimination of irreversible sorption from the equation could cause incorrect result during curve fitting solution.

Generally applicable result of my research work is the comparison of breakthrough curve experiment and impulse test based on parameter sensitivity analysis. According to my result I proved the impulse test has higher parameter sensitivity on investigated transport parameters which could ensure more accurate result during curve fitting solution.

The investigation of Lake Rudabánya from aspect of limnology enlarges our knowledge about the lakes of Hungary. The evaluation of lake as a mine pit lake has international importancy because generally the mine pit lakes can be found on the mine sites where the phenomenon of AMD is significant. This phenomenon cannot be found on Rudabánya that is the reason why the limnological properties of lake are interesting.

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IV. My publications related to the topic

1 Tóth Márton

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Doktoranduszok Fóruma Műszaki Földtudományi Kar Szekciókiadvány: Kiadó: Miskolci Egyetem Tudományszervezési és Nemzetközi Osztály. Konferencia helye, ideje: Miskolc, Magyarország, 2011.11.08 Miskolc: pp. 111-116.

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2 Tóth Márton

Nehézfémek migrációjának vizsgálata felhagyott bányatérsegekben.: Műszaki Tudomány az Észak-Kelet Magyarországi Régióban 2012 Konferencia. Szolnoki Főiskola, Szolnok, 2012. május 10. Konferencia kiadvány, Kiadja: Debreceni Akadémiai Bizottság Műszaki Szakbizottsága. ISBN 978-963-7064-28-9. Szekesztette: Pokorádi László. Debrecen 2012.

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3 Tóth Márton, Kovács Balázs

Modeling of water quality change in catchment of abandoned ore mine site

In: Zoltán Gribovszki, Kamila Hlavčová, Péter Kalicz, Silvia Kohnová (szerk.)

Egyéb szerzőség: Péter Csáfordi (sajtó alá rend.);

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Lektorált.

4 Tóth Márton

A NEHÉZFÉMEK MIGRÁCIÓJÁNAK MODELLEZÉSE A RUDABÁNYAI MEDDŐHÁNYÓKBAN

In: Szücs Péter, Tompa Richárd (szerk.)

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5 Tóth Márton, Kovács Balázs

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In: Zákányi Balázs, Faur Krisztina Beáta (szerk.)

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7 Tóth Márton, Kovács Balázs, Czinkota Imre

SZEKVENCIÁLIS EXTRAKCIÓS VIZSGÁLATOK RUDABÁNYAI MEDDŐHÁNYÓ MINTÁKON

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Könyvrészlet/Konferenciaközlemény/Tudományos

8 Tóth Márton, Móricz Ferenc, Kovács Balázs
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