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Heavy metals removal in biological wastewater treatment dependent on process parameters

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ABSTRACT

The aim of this study was to analyse the research data collected over a 5-year period, observing the interrelations between the process parameters of an operating wastewater treatment plant and the biosorption and adsorption of heavy metals in activated sludge. In addition, the following daily key parameters of the activated sludge process were determined: hydraulic retention time (HRT), mixed liquor suspended solids (MLSS), sludge retention time (SRT), pH, sludge volume index and dissolved oxygen. In the case of HRT, an inverse linear interrelation was found: the longer the HRT, the less heavy metals accumulated in the activated sludge. The SRT impact assessment revealed that the accumulation of all the metals in the activated sludge was at the highest for SRT periods of 15–19 d. The examination of MLSS concentrations in the range of 3,500–6,000 mg/L indicated that the accumulation in the activated sludge decreased in the given range.

Keywords: Heavy metals; Heavy metals accumulation; Heavy metals in activated sludge; Heavy metals in biological wastewater treatment; Tertiary treatment

1. Introduction

Heavy metals are defined as metals with a density of more than 5 g/cm^3 that can be toxic to living organisms at low concentrations [1]. Heavy metals are natural components of the Earth's crust, with some of them serving as necessary trace elements for organisms in their metabolic process [2,3].

Water pollution caused by heavy metals has become a serious environmental problem, especially due to accumulation. Scientists have observed the accumulation of heavy metals in the bottom deposits of the receiving waters of wastewater treatment plants (WWTPs) and their bioaccumulation in living organisms [4]. The quantities of heavy metals released into the environment depend on local conditions, such as the types of industry in the area, habits of the residents, sewage systems and the capability of a WWTP to remove heavy metals [5].

Heavy metals are persistent in the aquatic environment and therefore pose a hazard to living organisms. Each metal has its own unique characteristics and consequently, the specifics of toxicity vary by elements. However, those of the highest concern include water-soluble compounds, as they reach living organisms faster [6]. It is important to know the particular nature of metals and the associated risks, since each metal behaves differently in the environment and the hazard may vary in different environmental conditions, arising from, for example, the solubility at different pH levels. In the tissues of living organisms, heavy metals react strongly with amino acids of proteins, especially with cysteine [7,8]. Several heavy metals become toxic only in large quantities and act as important catalysts in many enzymatic functions. There is no enzymatic activity without a catalyst.

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However, hazards will occur when the metal concentration in organism becomes high and an enzyme is involved, which plays an important role in the cleavage of casein, gluten or other nutrients. Nutrients that are normally nontoxic when undigested, become toxic to organism. The metals can also cause a formation of free radicals, which by turn will destroy the cell membrane, as a consequence of the oxidation of fatty acids [9].

For the wastewater operators, the removing of heavy metals from wastewater is challenging, since the compliance is required for both the effluent and the sludge. The treatment processes currently in use are not capable of removing heavy metals with the required efficiency [10,11]. Fig. 1 shows the route of heavy metals from their primary source to the WWTP and further into the environment.

The emissions of heavy metals released to the aquatic environment may be reduced at different primary sources. For example, several cosmetic products contain heavy metals, which finally end up in sewerage (Fig. 1). The study by Ullah [12] shows that all the 15 cosmetic products analysed also contained all the heavy metals selected for this research. Zn and Cu may also reach the environment through storm water. In the Nordic countries, most of the houses have a galvanised steel roofing that should protect against corrosion; however, some of the Zn ions are carried out with storm water and released either to the WWTP or straight into the environment. The study by Charters [5] on the storm water runoff from zinc and copper roofing indicated that the concentrations varied from 397 to 1,970 μg/L for Zn and from 1,663 to 7,860 μg/L for Cu during the research period. Zn is also used in the process of vulcanising car tyres, whereas Cu is used in the construction of roofs and facades, but also in the drinking water piping in buildings, where Cu ions are released in case of an aggressive water [5,13]. The requirements for releasing heavy metals into receiving waters are very strict, and many WWTPs face serious problems meeting these requirements. In Estonia, the effluent released into receiving waters must comply with the limit values set for the heavy metals, examined in this research, as follows: 50 μg/L for Cr, 34 μg/L for Ni, 15 μg/L for Cu, 50 μg/L for Zn, 10 μg/L for As and 14 μg/L for Pb, with these limits often being lower than the natural background [14].

Some promising technologies for the removal of heavy metals include adsorption with activated carbon, ion exchange, coagulation, and electrodialysis that can be used at small flow rates [15,16]. Also, there are some more studies under way on using, for example, biological adsorbents, such as algal, fungal and bacteria. All these technologies can be applied to remove heavy metals, however, for the large municipal WWTPs only few methods, such as using activated carbon are applicable [17,18].

The removal of heavy metals from the wastewater in the activated sludge process is a result of different processes, such as adsorption and biosorption [11,18]. A part of heavy metals adsorbs onto the activated sludge flocs during physical and chemical processes, while the other part is aggregated biologically as a result of metabolism in microorganisms (biosorption). However, it is not clear yet how this is affected by different process control parameters [3,18,19].

The aim of this study was to analyse the research data collected over a 5-year period, observing the relations between the process parameters of an operating WWTP and the accumulation of heavy metals in activated sludge, in order to give an operational input on how to influence the process. The research will become relevant, when an engineer starts to design a technology for the removal of hazardous substances for the WWTP. If the beneficial use of sludge is desired for nutrient recovery in land, it is important for the sludge to contain minimum amount of heavy metals, with metals being removed from the wastewater during tertiary treatment. If the sludge is incinerated, the majority of heavy metals should accumulate in the sludge, thereby reducing the need for any tertiary treatment of wastewater.

2. Materials and methods

The data used in the research are obtained from a municipal WWTP with the load of about 450,000 p.e. The average incoming flow rate at the WWTP in the research period was

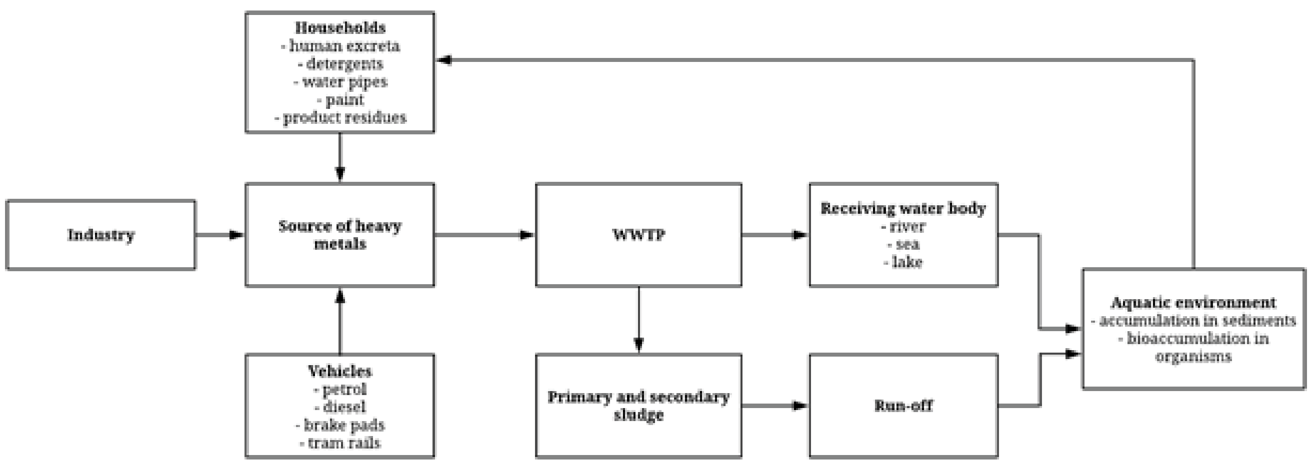

Fig. 1. The route of heavy metals into the environment [11], [12].

Table 1
The influent data of the WWTP under examination (n = 1,820)

Parameter	mg/L
BOD_7	484
COD	190
Suspended solids	349
P_{tot}	6.32
N_{tot}	49.64

131,297 m^3/d. The treatment process consists of mechanical treatment with screens, sand traps and primary tanks, and biological treatment carried out by means of an activated sludge process. 33% of the aeration tank is anoxic for the purposes of denitrification, and because of low carbon levels methanol is injected. Additional denitrification filter is used after the clarifier. Phosphorus is removed chemically, by injecting coagulant $Fe_2(SO_4)_3$. The raw sludge and wasted activated sludge are stabilised anaerobically in mesophilic digesters. Average influent data for the 5-year research period is presented in Table 1.

The samples of heavy metals required for the analysis were collected over a 5-year period (2011–2016), where every 2 weeks, samples averaged over 24 h were taken into a plastic container from the influent and effluent of the aeration tank by using the Endress-Hauser ASP-Station 2000 automatic composite sampler. Taking of the averaged samples was dependent on time: in every hour, a 200 mL wastewater sample was collected. In total, n = 118 samples of heavy metals were collected and the operating parameters for the respective days were logged. The total content of heavy metals was determined by an accredited laboratory, following the ISO 17294-2:2003 standard (application of inductively coupled plasma mass spectrometry).

The process parameters were logged automatically, by using the Vera software designed for WWTPs, which recorded the daily and hourly data from the operation of the whole WWTP. The following parameters of the activated sludge process were used in the research: hydraulic retention time (HRT, Eq. (1)), mixed liquor suspended solids (MLSS, applying EVS-EN 872 Solids Standard), sludge retention time (SRT, Eq. (2)), pH and dissolved oxygen. In addition, some other process parameters were analysed to determine whether the results of a particular measuring day could be affected by some exceptional event, such as rainy weather, the number of operating sludge centrifuges and technical disturbances in the process.

$$\mathrm{HRT}(\mathrm{h}) = \frac{V_{\text{aeration tank}}\ \mathrm{m}^3}{Q_{\text{influent}(h)}\ \mathrm{m}^3/\mathrm{h}} \tag{1}$$

$$\mathrm{SRT}(\mathrm{d}) = \frac{V_{\text{aeration tank}}\ \mathrm{m}^3 \times \mathrm{MLSS}(a)\,\mathrm{kg/m}^3 + Q_{\text{influent}(d)}\mathrm{m}^3/\mathrm{d} \times \mathrm{SS}(i)\,\mathrm{kg/m}^3}{V_{WAS}\mathrm{m}^3/\mathrm{d} \times \mathrm{MLSS}(w)\,\mathrm{kg/m}^3 + Q_{\text{influent}}\mathrm{m}^3/\mathrm{d} \times \mathrm{SS}(e)\,\mathrm{kg/m}^3} \tag{2}$$

where $V_{\text{aeration tank}}$, total volume of aeration tank (anoxic + aerobic), m^3; $Q_{\text{influent }(h)}$, influent flowrate of aeration tank, m^3/h; $Q_{\text{influent }(d)}$, influent flowrate of aeration tank, m^3/d; SS(i), suspended solids in the influent of aeration tank, kg/m^3; MLSS(a), mixed liquor suspended solids in aeration tank, kg/m^3; V_{WAS}, flowrate of the removed wasted activated sludge from clarifier, m^3/d; MLSS(w), mixed liquor suspended solids in wasted activated sludge, kg/m^3; SS(e), suspended solids in the effluent of clarifier, kg/m^3.

The data were analysed, using MS Excel and GraphPad Prism software, applying the following methodology:

- The mass of heavy metals removed in the activated sludge process (M_{removal}) was identified by using the following equations:

$$M = C_{\text{HeM}} \times Q_{\text{influent}}(d) \tag{3}$$

$$M_{\text{removal}} = \left(M_{\text{influent}} - M_{\text{effluent}}\right) \tag{4}$$

where M, mass of heavy metal, mg; C_{HeM}, concentration of heavy metal in influent or effluent, mg/m^3; $M_{\text{influent or effluent}}$, total mass of heavy metal in influent or effluent, mg.

- HRT and the corresponding M_{removal} were sorted and expressed as a linear function, where HRT is placed on the x-axis and the mass of heavy metals removed (in mg) on the y-axis. The HRT examined remained between 5 and 18 h.
- In order to identify the relations between SRT and the removal of heavy metals, a 5-year SRT and the corresponding M_{removal} were sorted by length. The data were presented on a graph with SRT placed on the x-axis and M_{removal} on the y-axis. SRT intervals from 11 up to 24 d, used for operating during this period, were examined.
- In order to identify the relations between MLSS and the heavy metals, MLSS concentrations in the aeration tank were sorted (on the x-axis) and the corresponding M_{removal} (on the y-axis). The rounded MLSS values analysed were in the range of 3,500–6,000 mg/L.

3. Results and discussion

3.1. Concentrations of heavy metals in the WWTP

Fig. 2 shows the average concentrations of six heavy metals in the influent and effluent of the WWTP examined in 2011–2016. The highest influent concentrations were 108.27 μg/L for Zn and 45.18 μg/L for Cu. The reasons for this are partially explained also in Fig. 1. As the examined WWTP is partly receiving also storm water, the main sources of Zn include zinc roofing and street railings. Cu also originates from roofing and from widely used water pipes [5,20]. In the research period, the removal efficiency was 84.7% for Zn and 82.7% for Cu. Similar results were also observed by Luo [21] in the study where 94.1% of Cu and 75.3% of Zn were removed in the course of biological treatment under laboratory conditions. Hereby, the term 'removal' refers to the removal of heavy metals from the aqueous phase, that is, since the metals persist, the percentages cited earlier refer to the fact that in the wastewater treatment process, the difference between the influent and effluent parameters is due to the adsorption of Zn and Cu in the activated sludge or their biosorption in micro-organisms. As and Ni were the

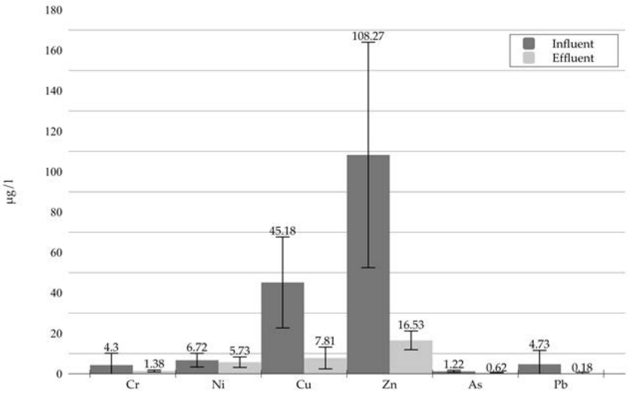

Fig. 2. The average concentrations of heavy metals during a 5-year period in influent and effluent of the wastewater treatment plant under examination (n = 118).

least-decreasing metals in the course of the examined wastewater treatment process, with a removal efficiency of 49.2% and 14.7%, respectively. These were also the elements with the lowest concentrations.

The study by Chipasa found that the higher the concentration of a heavy metal in the influent, the greater the removal efficiency, which was also confirmed in this research. In addition, the removal of heavy metals depends on their water solubility at different pH values. In this research, the pH value varied in the range of 7–9 [2]. Different studies point out that the better the water solubility of a heavy metal, the greater the biosorption and the smaller the physical adsorption [22,23].

3.2. Relations between HRT and the removal of heavy metals

HRT is one of the key parameters in the activated sludge process: the longer the HRT, the more time micro-organisms have to aggregate the nutrients. This study allowed for a reliable analysis of HRT in the range of 5–18 h. Single days where the retention time was shorter or longer than the given range were not considered, as the number of these days was insufficient for the corresponding data analysis. The results are presented in Fig. 3.

Fig. 3 shows a strong linear relation between HRT and the removal of heavy metals. The strongest correlation coefficient was found for Cu where R^2 was 0.9263, and the smallest given parameter of 0.7206 was found for Pb, respectively [24]. This correlation is good, considering how many different factors affect biological wastewater treatment and that any R^2 over 0.7 is considered as a strong linear relation.

The research shows that the retention time mostly affects the removal of Cu. In the case of Cu, it was found that the removal rates for the minimum and maximum retention times examined were 7,399.4 and 1883.7 mg Cu/d, respectively, which makes a 74.5% difference in the removal efficiency (Fig. 3). The removal of Pb was the least influenced by the retention time. During the minimum and maximum retention times, 568.9 and 329.1 mg Pb/d, respectively, were adsorbed in the sludge, which makes a 42.2% difference in the removal efficiency. The respective differences between the minimum and maximum removal efficiencies were 70.6% for Cr, 64.9% for Ni, 62% for Zn and 70.5% for As. There are no clear answers to such linear relation in publications to date, but according to different studies it can be explained by an inhibition of biological treatment caused by different heavy metal compounds. Malamis found in his research that already small concentrations of heavy metals (inhibition up to 49%, concentrations of heavy metals varied from 10.2 to 411.1 μg/L) cause significant inhibition of the heterotrophic biomass activity. Similar results were also found by Feng [25–27]. In other words, longer retention time inhibits the metabolism in micro-organisms and decreases biological biosorption [28,29]. Regarding Cu and Zn, Özbelge found an inverse relation in the tests conducted in laboratory, where more heavy metals were aggregated in the sludge in the case of longer HRT. However, these tests used single heavy metals with different initial concentrations, referring to the

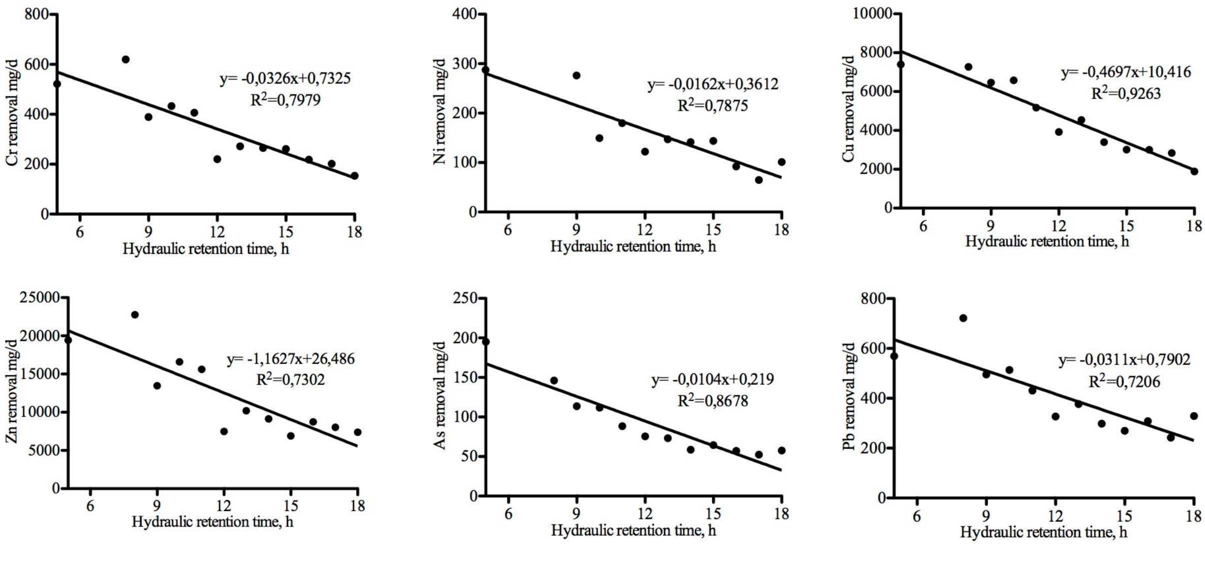

Fig. 3. The dependence of removal of the examined heavy metals on HRT.

possibility that this may differ from the combined removal of heavy metals in an operating WWTP [30].

3.3. Relations between SRT and the removal of heavy metals

The species composition of micro-organisms involved in the activated sludge process depends on the sludge age: a WWTP, designed for removing only carbon, operates at SRT of less than 5 d, and the SRT for a treatment plant designed for nitrogen removal is more than 10 d. It is well known that the biosorption of heavy metals by different micro-organisms and the adsorption of activated sludge flocs formed in different micro-organisms can vary. Some micro-organisms produce a substance during metabolism that will intensify the formation of activated sludge flocs and consequently, increase the adsorption of the suspended solids. The greater the adsorption, the more of heavy metals will be removed from the wastewater.

The WWTP under examination removes organic matter, N, as well as P from the wastewater and therefore, SRT of more than 10 d is applied. Fig. 4 presents the dependence of removal of heavy metals on SRT.

The dependence detected for SRT was not as strong as for HRT, however, it can be observed with all the examined metals that the highest amount of heavy metals are removed from the sludge that is 14–18 d old. The greatest dependence can be observed in the case of Cr, which clearly shows that the removal of Cr increases with the increasing sludge age and starts to drop again after day 17. This dynamic can be explained by micro-organisms, characteristic to given sludge age, and by endogenous respiration caused by long SRT, causing the redissolution of aggregated heavy metals to increase. Similar hypothesis was also formulated by Gulyás [18]. Study by Ong concluded that the longer the SRT, the more the combined toxicity of heavy metals will start to inhibit biosorption and biological treatment [31]. The review by Dhokpande found that the highest rate of heavy metals removal was achieved at the sludge age of 12 d. However, as an operating WWTP is a very complicated system, it is not possible to give a definite answer[32]. It can also be seen in Fig. 4 that the removal capacity begins to increase starting from SRT of 23 d, but since the longest SRT at the WWTP under examination was 24 d, the given dynamic could not be clearly proven.

3.4. Relations between MLSS and the removal of heavy metals

While the species composition of the micro-organisms depends on SRT, the number of micro-organisms in the aeration tank depends on MLSS. The study examined rounded concentrations of MLSS in the range of 3,500–6,000 mg/L and the removed quantities of heavy metals in this range. The linear relations between the analysed data are presented in Fig. 5.

Fig. 5 shows that MLSS mainly affected the removal of As, where the difference in the removal efficiency was 76.6%, which is also characterised by a strong R^2 of 0.7252. Similar dependence was also found for Zn and Cu, where the difference in the removal efficiency was 38.7% and 32.7%, and R^2 0.6515 and 0.7438, respectively. The smallest dependence was found for Ni, where the difference between the removal rates at the minimum and maximum MLSS was 3.5% with no linear relation identified either. The possible causes for the reduced removal of Ni are presented in Section 3.1. However, the removal efficiency for all the examined heavy metals decreased when MLSS started to increase. The similar results were confirmed by Hammaini and Wu in their laboratory tests, where the sorption of heavy metals decreased with the increasing MLSS, explained by the screen effect where bigger biomass starts hindering the sorption [33,34].

4. Conclusions

With the use of heavy metals in our products, we must bear in mind that they will not disappear anywhere but

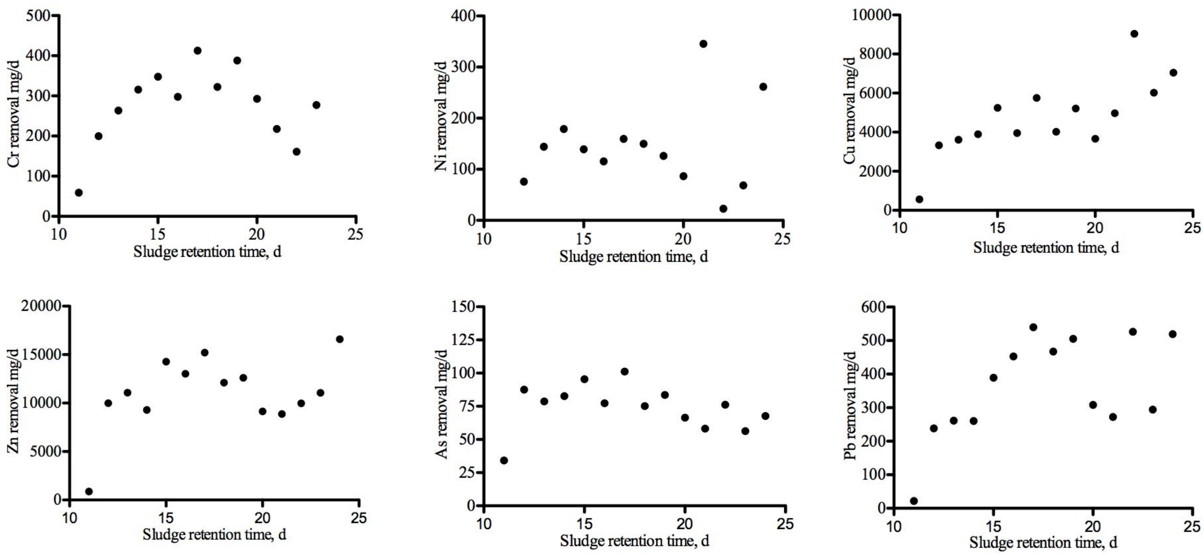

Fig. 4. The dependence of removal of the examined heavy metals on SRT.

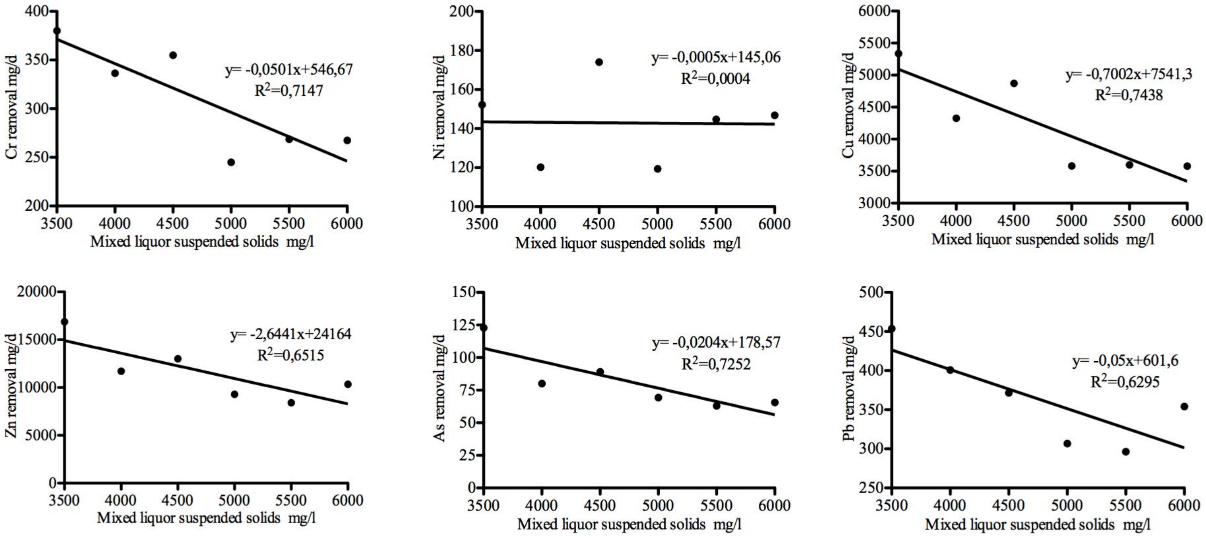

Fig. 5. The dependence of removal of heavy metals on MLSS.

will remain circulating in the environment. Therefore, the simplest way to decrease the quantities of heavy metals is not to use them in products. Various treatment processes that have been developed do not solve this problem; they simply store it in other places, such as in sludge or on landfills, where it is consequently often necessary to clean the leachate. In other words, heavy metals already removed must again be retrieved from the environment.

This research showed that the accumulation of heavy metals in activated sludge could be influenced by the key parameters of an activated sludge process. Since the effluent from the WWTP conformed to national standards during the whole research period, the obtained results could be used for operating a WWTP. However, it should be clear at which point we wish to remove the heavy metals – from the sludge or from the aqueous phase during tertiary treatment. Further studies are needed to estimate how the increased concentrations of heavy metals in the activated sludge affect the biological treatment in general, and whether any combined effect of heavy metals exists, which would inhibit the removal of organic matter and N.

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