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Evaluation of a pilot plant for a secondary treatment of mining effluents

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ABSTRACT

Mining effluents can often contain heavy metals such as Lead (Pb), Zinc (Zn), Iron (Fe), Manganese (Mn), Cadmium (Cd), Arsenic (As) and if left untreated can cause damage to the local aquatic environment. In this study, it is demonstrated the operation of a 9 m^3/h pilot unit (PU) installed in Greece. It consists of a pH regulation and oxidation stage, followed by filtration to a catalytic multimedia filter, activated carbon filter, and a reverse osmosis (RO) unit. PU is fed by the existing pretreatment installation outlet. Our results prove that PU installing improved the water quality of water discharge, so that the final concentrations of dissolved metals to be even lower than the legislation limits for potable water. The removal of Fe, Mn, and Zn was 90%–100%, Cd was 80%–90%, while Pb and As was <80%. Operational data demonstrated very good removal efficiency of the Filtration Stage so that RO stage can be omitted. PU successful application enables design to be applied covering the complete installation needs. The operation of PU is simple, automatic, and constant. It is environment-friendly and economically viable in terms of fixed and operational cost.

Keywords: Mine; Effluents; Heavy metals; Filtration

1. Introduction

Mining effluent takes up a very large proportion of the total volume of waste produced in the European Union (approximately 30% in 2012) [1–3]. The presence of heavy metals such as Lead (Pb), Zinc (Zn), Iron (Fe), Manganese (Mn), Cadmium (Cd), and Arsenic (As) are of major concern due to their nonbiodegradability. Furthermore, the accumulation in the living organisms of the local environments causing severe health problems in animals, plants, and humans such as cancer, kidney failure, metabolic acidosis, oral ulcer, and renal failure [4].

The main methods used for the heavy metal removal from wastewater are precipitation, oxidation, ion-exchange, and adsorption [5]. However, membrane processes have shown a great promise due to their efficiency [6–8].

Except reverse osmosis (RO) other membrane processes that have been tested for metal removal are ultrafiltration (UF) and nanofiltration (NF). While NF and RO require a medium to high pressures, UF can operate to low transmembrane pressures, but the footprint of the treatment plant should also be considered when an industrial scale application is designed [9].

Coagulation and flocculation followed by sedimentation and filtration are used as well. Different treatment methods are flotation and electrochemical treatment [1,9]. Floatation is a pH dependent process with a medium to high efficiency at metal removal, while the high capital cost for electrochemical treatment is the major drawback for industrial scale applications [9].

In our study, the effluent produced at the mining site "Olympias" and "Madem Lakkos," privately owned by Hellas Gold S.A., was examined. Wastewater is discharged due to the treatment of the gold bearing arseniferous pyrite

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concentrate [10] and it is directed to an existing wastewater treatment plant which consists of primary physicochemical processes. The scope of the pilot unit (PU) was to improve heavy metal removal following the primary treatment so that the final effluent satisfies even lower limits than those enforced by the environmental permit. For this goal a process was designed based on the oxidation of heavy metals with modified catalytic sand filters (CSFs) and granular activated carbon (GAC) filters. The choice of the CSF and GAC as a main processing step was based on their heavy metal removal efficiency [4,11–13]. Its main advantages compared with other process are: (1) minimum amount of discharge, (2) very limited use of chemicals, (3) small installation space requirement, (4) low electricity consumption, and (5) absence of any odor.

The basic theory behind the overall procedure is the adsorption of the heavy metals on the GAC [14]. Although other sorption materials are emerging, activated carbon remains the most popular and widely used method and its efficiency counteracts the initial capital cost for its establishment [15].

The operation of the installation is simple, automatic, and constant, requiring minimum time for daily check. It is financially attractive in terms of fixed (CAPEX) and operating cost (OPEX) especially considering that the CSF-activated carbon filters (ACF) filtration stage can be used alone in order, to achieve very good heavy metal removal efficiency. RO is used as a final optional polishing treatment after the activated carbon filtration stage.

2. Materials and methods

The existing waste water treatment plant (WWTP) at Site 1 "Olympias" and Site 2 "Madem Lakkos" are consisting of a primary treatment stage where the removal of turbidity and bulk contaminants is achieved by adding lime emulsifier. The PU was installed as a secondary treatment stage and during its monitoring the metal concentrations of the collected samples were determined by ICP (induced current plasma).

The data were treated separately for each metal. For segregation of the data from the outliers the H-spread method was incorporated. Based on the above method the first, second, and third quartile have been calculated, denoted as $Q1$, $Q2$, and $Q3$. The IQR value

$$\mathrm{IQR} = Q3 - Q1 \tag{1}$$

has been calculated and the dispersion of the dataset has been graphically represented by box plots (not included herein). Observations that found below the

$$Q1 - 1.5 \times \mathrm{IQR} \tag{2}$$

or above

$$Q3 + 1.5 \times \mathrm{IQR} \tag{3}$$

are considering as the outliers of the dataset. Following the trimming of the data, correlations between the metal concentrations to conductivity and pH have been made.

PU has three main stages

2.1. Pretreatment stage

The pretreatment step consists of: (1) Chlorination: Chlorination causes oxidation to iron, manganese, and heavy metal ions, which are converted into insoluble compounds and precipitate. The method is efficient to trace metals that can be oxidized to higher oxides [15]. Chlorination is applied by injecting a sodium hypochlorite (NaOCl) 12% solution at the inlet pipe of the aeration tank. (2) pH adjustment: Following chlorination, pH adjustment step is required to create an alkaline environment in the feed water, which improves the efficiency of the CSF filters.

The overall undergoing reaction mechanism with NaOCl is as follows:

$$2\mathrm{MO} + \mathrm{ClO}^- \rightarrow \mathrm{M_2O_3} + \mathrm{Cl}^-$$

where M is any catalytic metal [15].

Adjustment of pH is achieved by injecting a 50% caustic soda (NaOH) solution at the inlet of the aeration tank. (3) Aeration (oxygenation): In the aeration tank, mining wastewater goes through an oxidation stage to facilitate the removal of precipitated iron, manganese, and heavy metals. The typical aeration technique consists of a forced air flow, using blowers and diffusers to generate small air bubbles which improve the water–air contact.

2.2. Filtration stage

In this step filtration of the water is achieved by means of CSFs and GACs.

- CSFs: Downstream of the aeration tank, the water goes through the CSFs which retain various suspended particles, turbidity, Fe, Mn, and substances harmful to the treatment so that the Silt Density Index becomes less than 5. Water flows (at a suitable speed) through carefully preselected layers for the retention of contaminants on the surface of these materials. Filter layers are of different granulometry of quartz gravel (0.4–0.8, 1–2, and 3–5 mm), silica sand, pyrolusite (mainly consisting of manganese dioxide, $\mathrm{MnO_2}$) and anthracite as upper layer (0.8–1.6 mm). The method used for Fe and Mn removal from the water is a natural process which is based on oxidation by air (as mentioned earlier) combined with the use of chemicals commonly used in the treatment of drinking water, which is an advantage compared with the other processing methods. The tank is of industrial scale made from medium density polyethylene with a $d = 1.05$ m and filtration surface of 0.87 $\mathrm{m^2}$. The bed height is 1.5 m at 6.0 bar max operating pressure. The filtration velocity has been set at 10.5 m/h.
- GAC filters: After the CSFs water passes through the GACs, as a second filtration/adsorption stage for the removal of the remaining manganese and iron which passed through the CSFs and heavy metals such as arsenic, lead, zinc, chromium, cadmium, and arsenic. GAC quality has been selected to maximize GAC efficiency. The bed height is 1.5 m at 6.0 bar max operating pressure.

During the filtration through GAC, different parameters such as pH of the inlet water stream, dissolved oxygen, and flow rate were monitored and tested. To elucidate further the pH dependency, it was decided to focus on the removal of lead which had been found the most challenging particle to be removed. As it can be observed in Fig. 1, during the removal of Pb, two different removal mechanisms are occurring.

The first mechanism is the adsorption of the heavy metal cations on the surface of the GAC. During that step, the pH should be below 8 to avoid the oxide crystal formation. The second mechanism initiates at higher pH where oxides start to form and precipitate. At pH above 11, the major mechanism for lead removal is the hydroxide formation and precipitation [17,18].

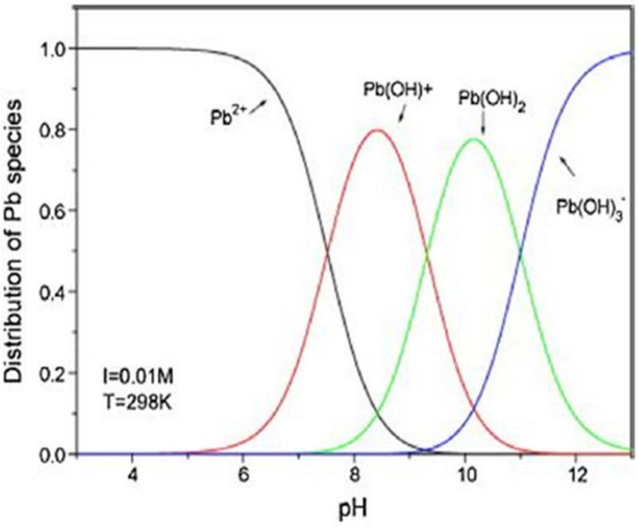

Fig. 1. Lead species over different pH values [16].

2.3. Polishing stage – RO

In RO, the water is fed to semipermeable membranes. RO has been designed as an optional process to ensure that even at the most challenging operational parameters permeate water will remain below the required limit. RO is suitable for removing very low concentrations of metals. In any case RO process requires a good pretreatment stage to operate without fouling as the water treated is wastewater. It has been designed to operate at 75% recovery ratio.

Two high-pressure vessels, PRO-8-450 from Protec Arisawa (Spain) were implemented with three membranes each. Membranes used are the CPA5-MAX from Hydranautics Nitto Denko (Japan) with 99.7% salt rejection each with 440 ft^2 active surface. At Fig. 2, it can be seen the flow diagram of the fully automated process which can be operated remotely as well. The pumps are Grundfos CRN series made of stainless steel wetted parts and have been sized according to the specifications of the project.

3. Results and discussion

The metal concentrations of the collected samples were determined by ICP. Initially the pretreatment step included oxidation only by aeration, but the Mn removal was not up to the desired limits. It has been observed that the process was more efficient when the dissolved metals in the effluent stream were oxidized by NaOCl dosing and the pH was regulated. In Figs. 3–17, concentration reduction of Mn, As, Cd, Zn, Pb, and Fe after the filtration, the RO stage of the PU, and after the introduction of NaOCl and NaOH to the pretreatment are shown. In most cases, metal concentration limits achieved project's requirements only by the implementation of filtration stage. RO polishing helped in the rare cases when the filtration effluent reached or slightly exceeded target limits for the metals concentration. More specifically, RO

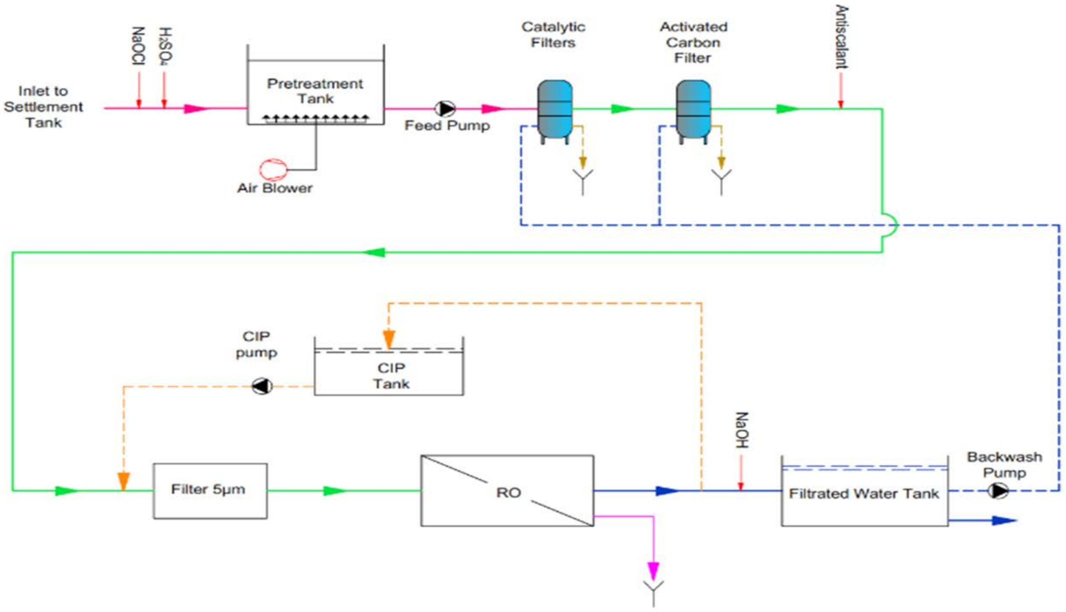

Fig. 2. Flow diagram of a pilot plant at Sites 1 and 2.

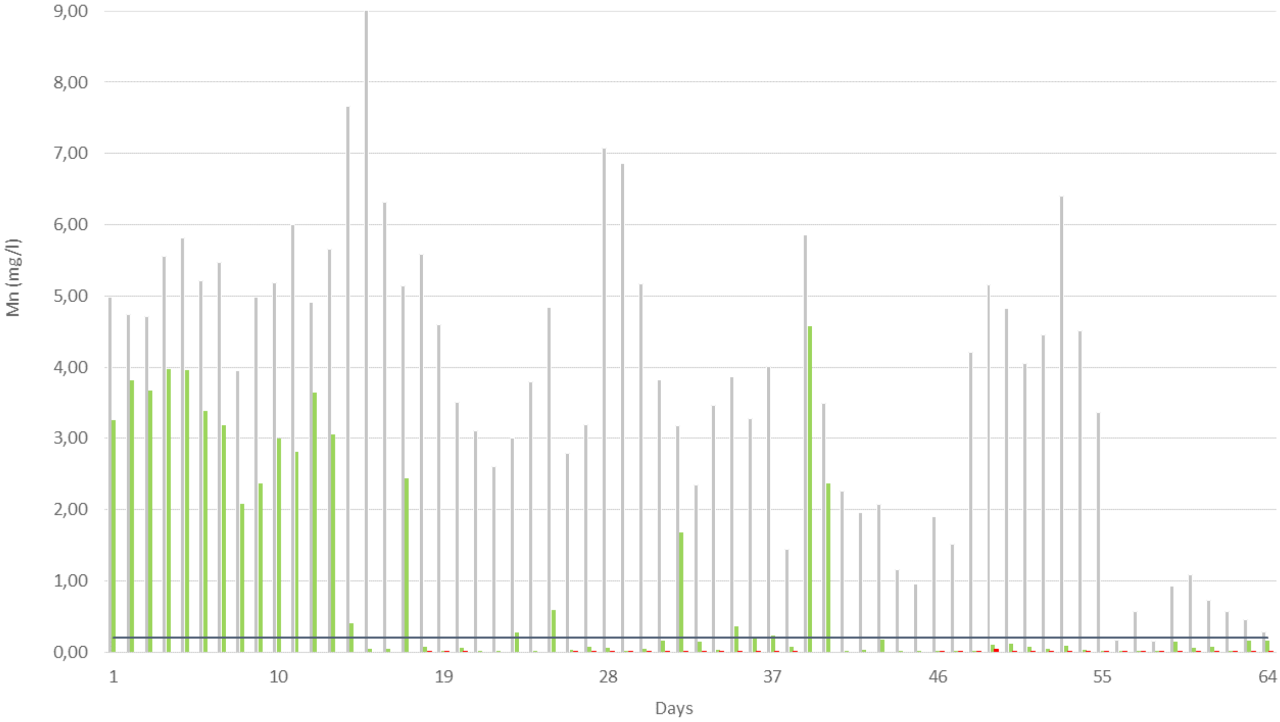

Fig. 3. Manganese removal at Site 1. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO (barely visible), and the blue line indicates the project limit.

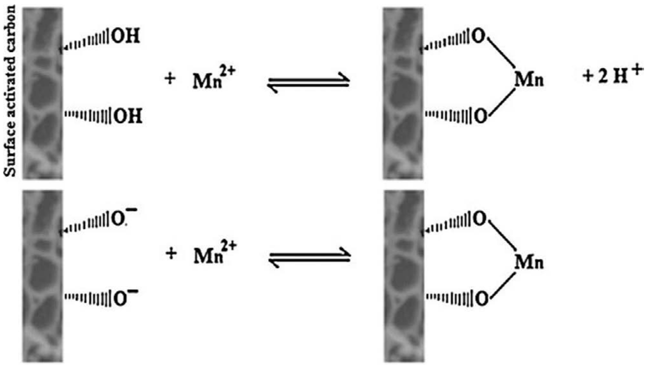

Fig. 4. Possible mechanism for Mn^{2+} removal by activated carbon [19].

unit was able to minimize the effluent concentrations of Mn and As but the impact on Cd, Zn, Pb, and Fe was negligible. This was anticipated since their concentration was in the ppb range where RO is not as effective.

As it can be seen in Fig. 3, during the first days there was a low removal efficiency of Mn^{2+} which later improved. The median feed was 3.907 ppm.

Following the increase of the pH a higher removal of Mn^{2+} was achieved. The effluent of the GAC was 0.08 ppm which is reflecting to 97.75% removal efficiency. That outcome corresponds to the expected behavior of Mn^{2+} ion, where the removal increases at higher pH. The contribution of RO installation following the GAC filters can be accounted for a further 93.75% removal. The possible mechanism for Mn^{2+} removal can be seen in Fig. 4.

The data were retrieved from the other site, in which the PU was installed, are very stable and below the permissible limit throughout the year of operation. Although the infeed Mn^{2+} concentration was lower, 0.387 ppm, the final effluent was 0.071 ppm (81.6% removal) and the contribution of RO installation can be accounted for a further decrease of 92.98%.

Concerning the arsenic (Fig. 6), the data from Site 1 exhibit a removal of 70.03%. The feed was measured at 30 ppb and the effluent was measured at 9.44 ppb, which results in a concentration below the legislation limit for potable water. The RO installation was accounted to a further removal of 84.86%, while the permeate had a concentration of 1.43 ppb.

The data from the Site 2 exhibit much higher infeed values which were measured at 52.91 ppb and the effluent were measured 25.67 ppb which also is below the project target values. The RO installation following the GAC filtration was accounted for a further decrease of 93.26% and a final effluent of 1.73 ppb.

In general, it has been noted that arsenic removal is decreasing as the pH is increasing. The observation is in accordance to literature references indicating similar results [20].

In Fig. 8, zinc removal at Site 1 was above 94% achieving the projects target. Relative to pH it has been noted to behave similar to the As cation, which shows that the formation of oxides is not contributing positively to the overall procedure.

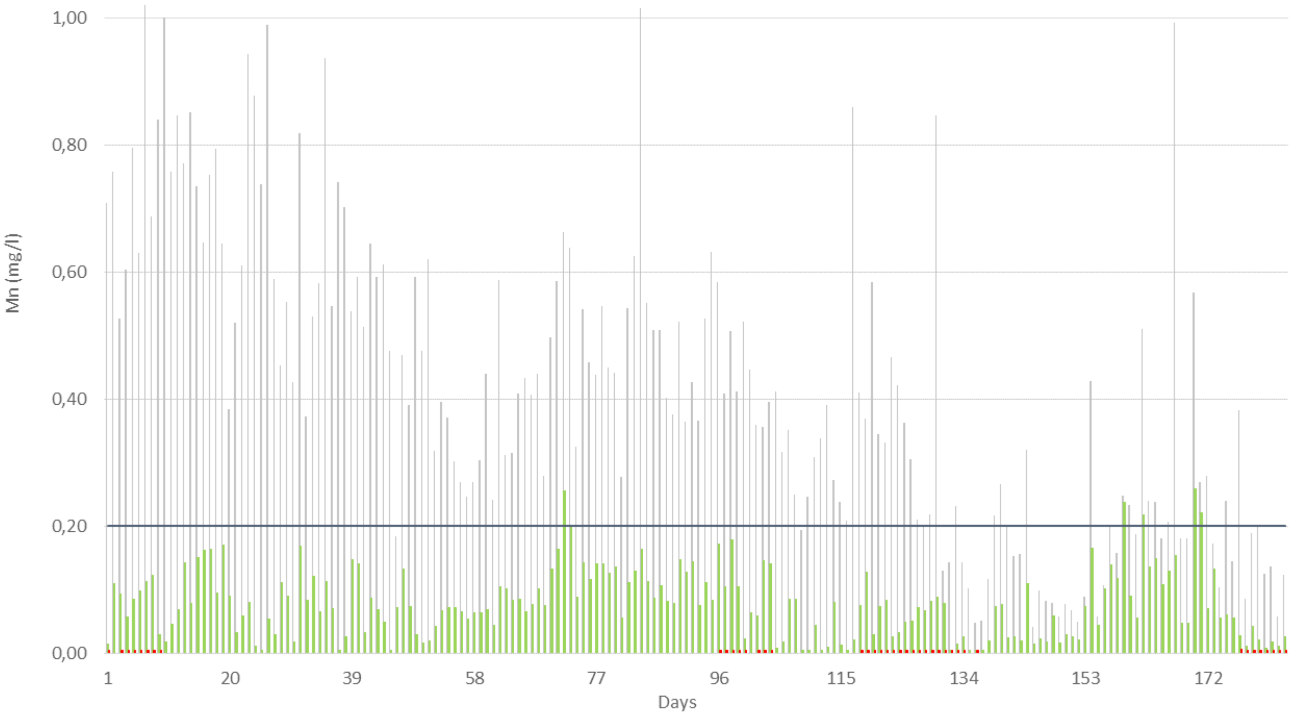

Fig. 5. Manganese removal at Site 2. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO (barely visible as the concentration is very low), and the blue line indicates the project limit.

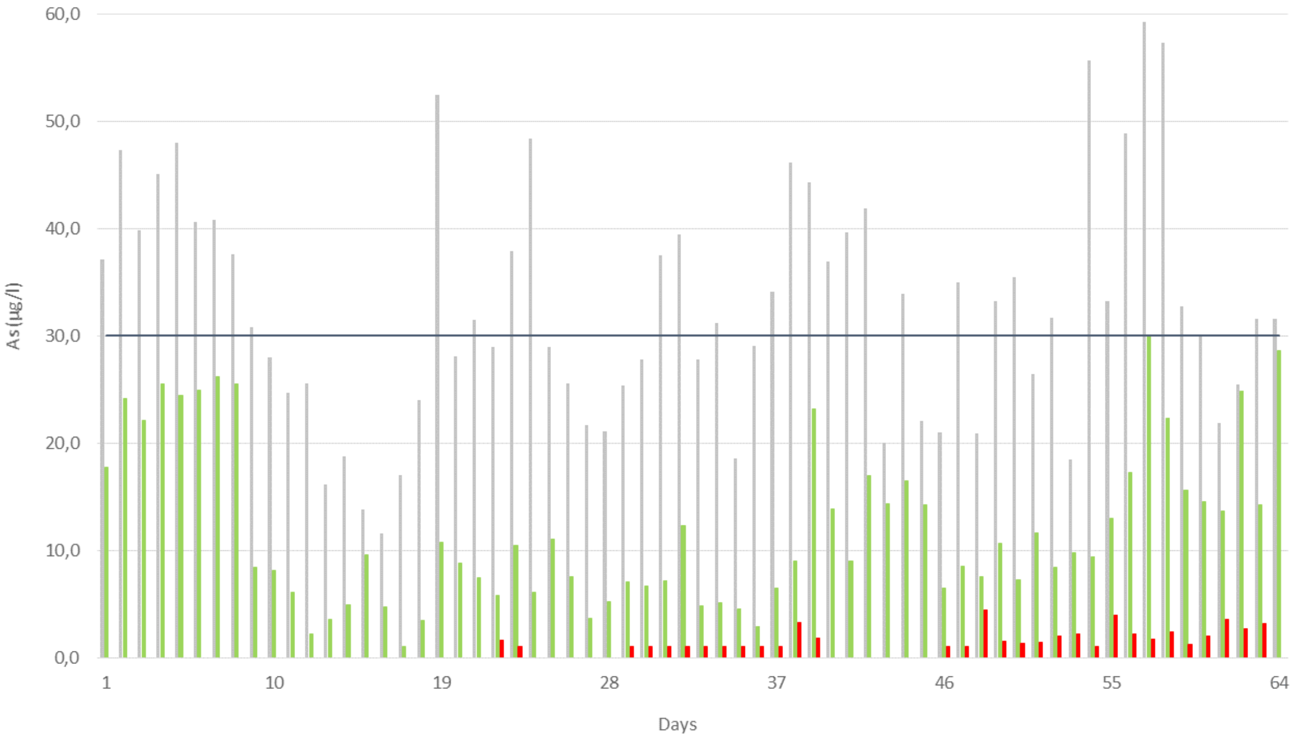

Fig. 6. Arsenic removal at Site 1. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the project limit.

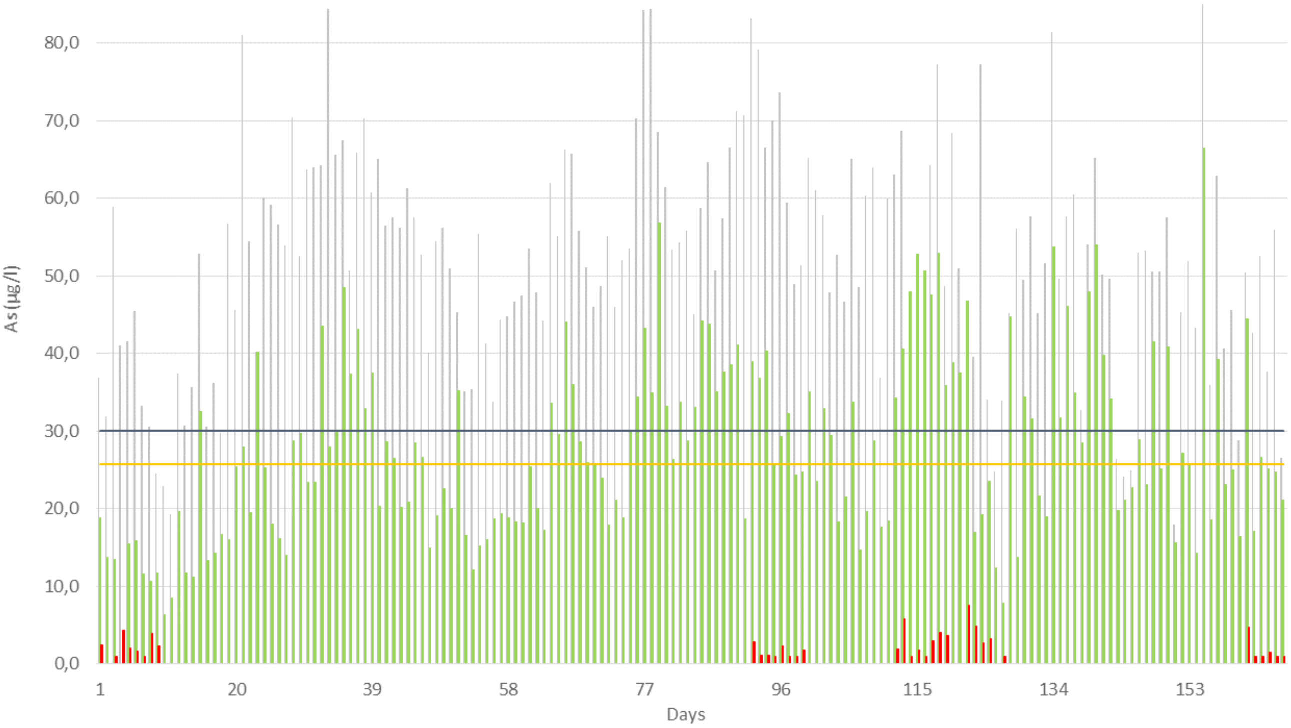

Fig. 7. Arsenic removal at Site 2. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the project limit and the orange line the mean annual concentration after filtration.

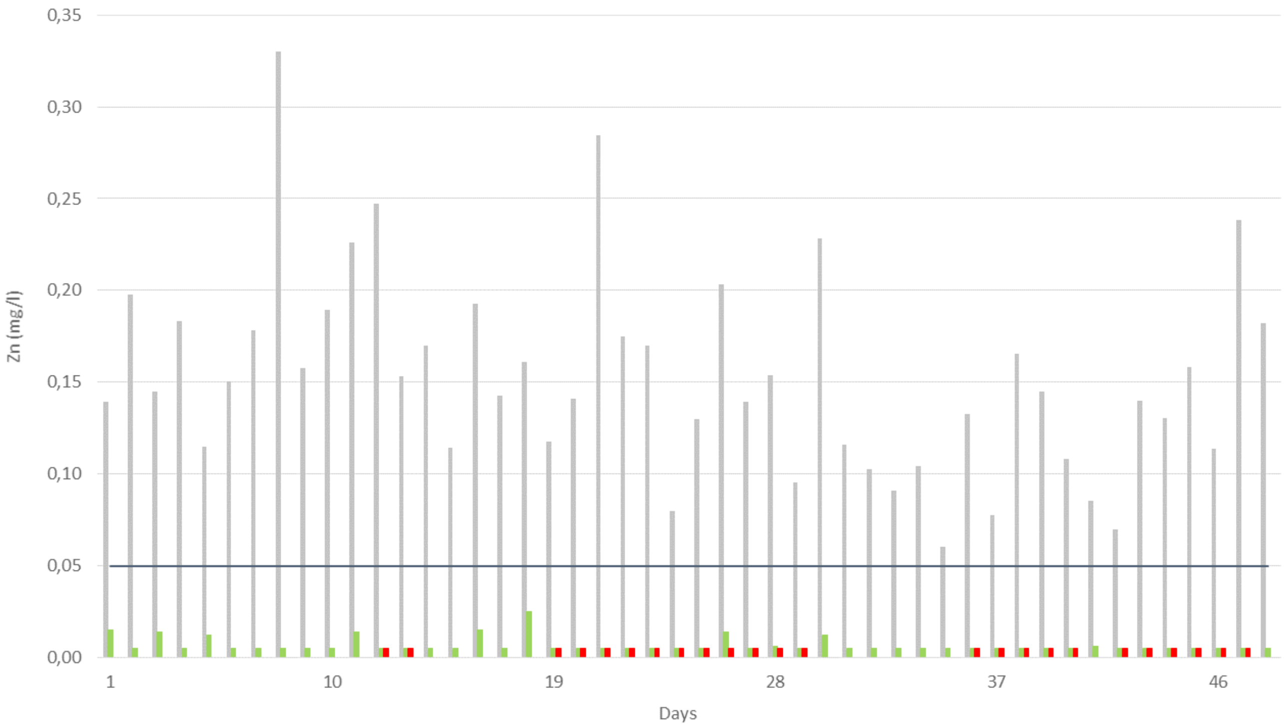

Fig. 8. Zinc removal at Site 1. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the project limit.

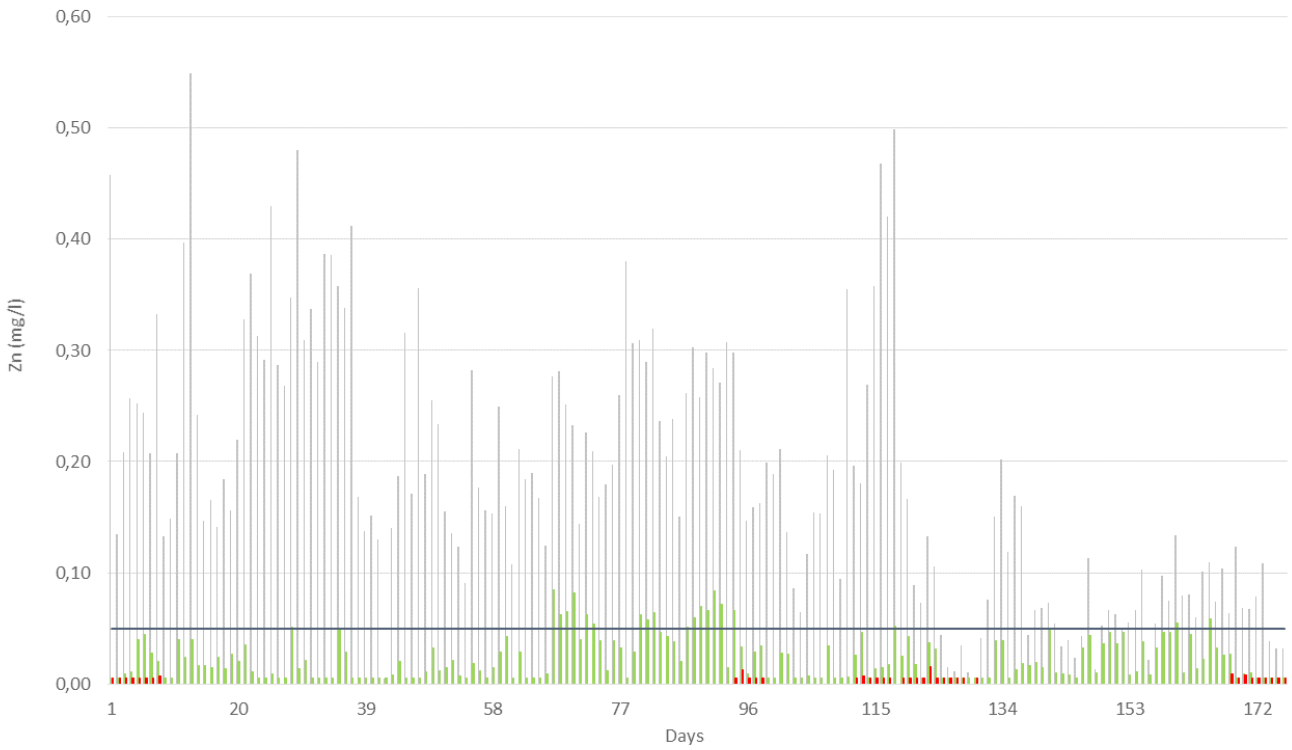

Fig. 9. Zinc removal at Site 2. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, the blue line indicates the project limit.

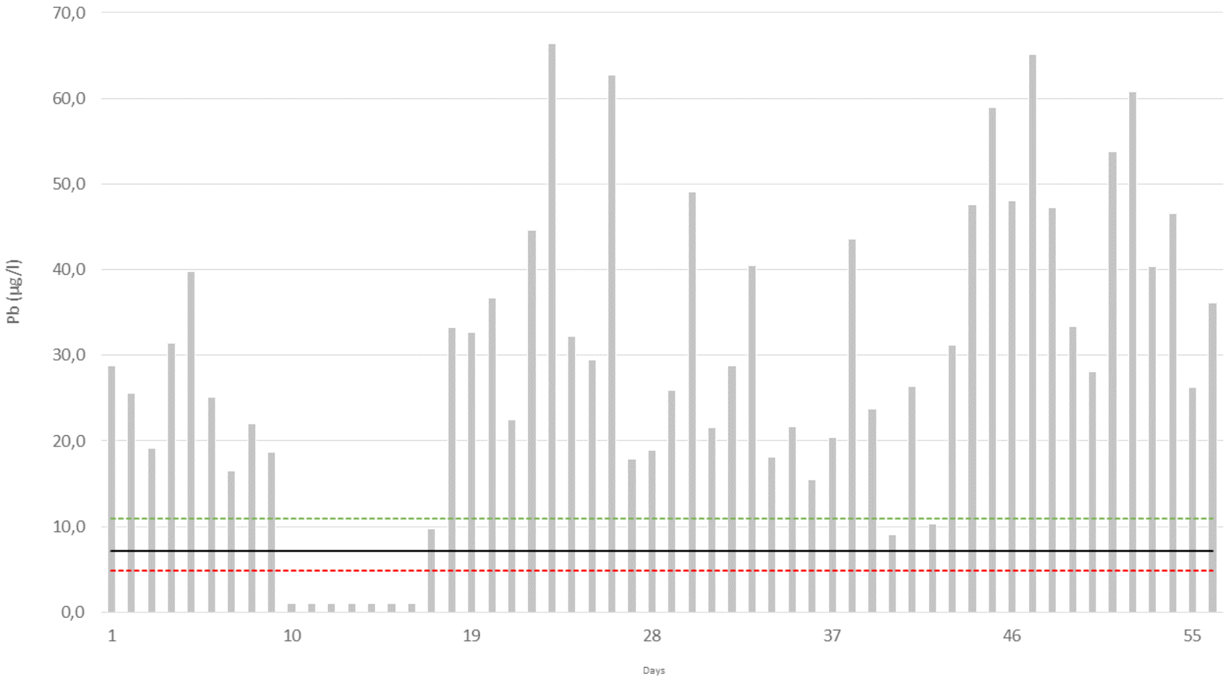

Fig. 10. Lead removal at Site 1. Grey lines represent the concentration before the filtration, the blue line indicates the target limit, the green line indicates the average filtration effluent concentration, and the red line indicates the after reverse osmosis.

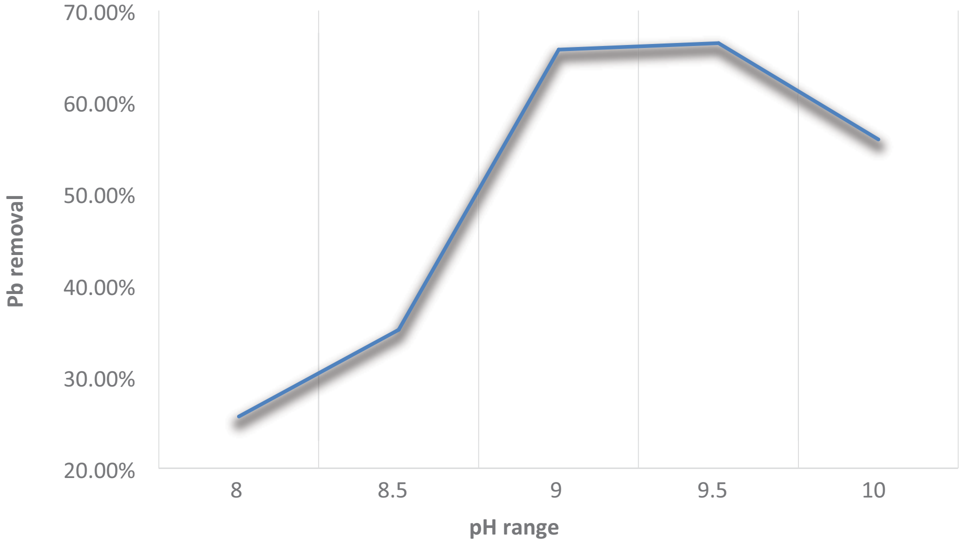

Fig. 11. Lead removal over different pH range values (site 1).

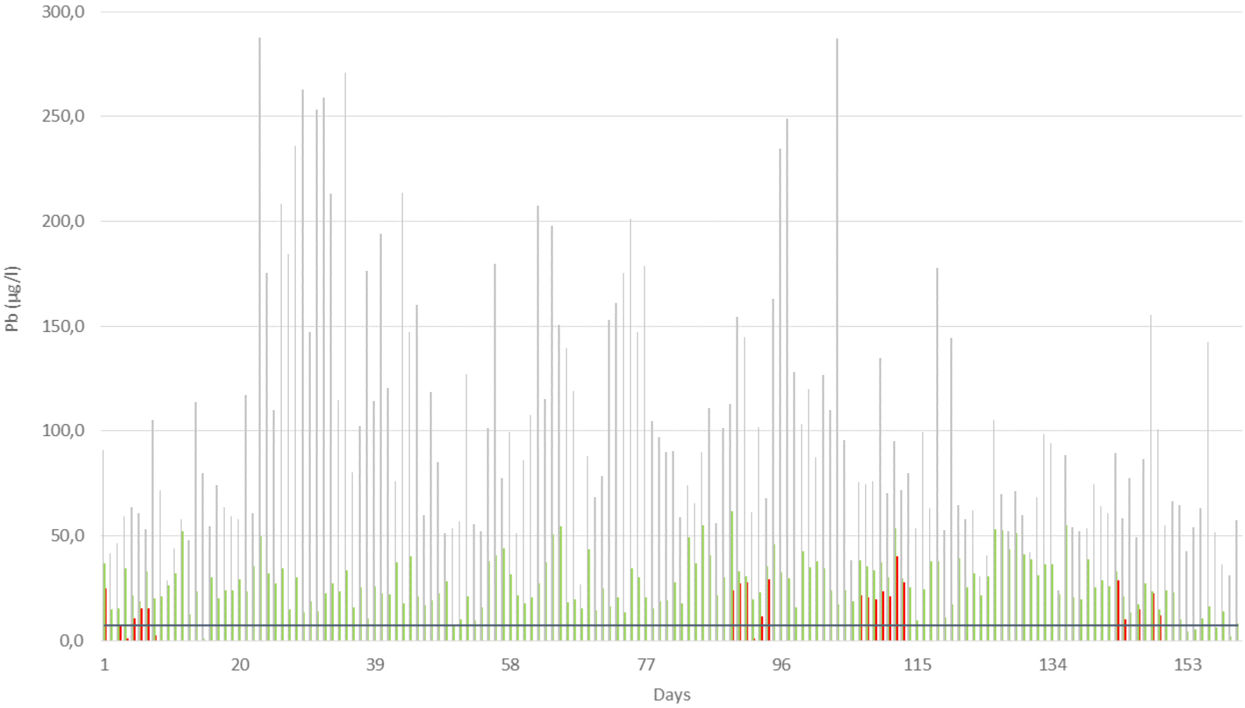

Fig. 12. Lead removal at Site 2. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the limit have been set as target.

On Site 2 the same tendency was observed, where the infeed values were 0.167 ppm and the GAC filtration step efficiently removed the 91.00%, whereas the final effluent concentration was 0.015 ppm. The RO installation was contributed to further 66.67% at least by producing a permeate with concentrations of zinc below the level of detection by ICP.

Lead removal was found to be the most challenging metal ion in terms of removal efficiency (Fig. 10). Pb removal efficiency is a very pH dependent process as referred in Section 2.2.

The infeed concentration was 27.21 ppb, while the effluent concentration was 10.93 ppm. The removal was 59.82%. Following the RO, the final concentration was 4.9 ppb, which is below the target limits that have been set on this project. The RO installation has contributed to a further 55.19% removal, a result that shows that a RO application without GAC pretreatment may not be the optimal procedure for treatment of Pb contaminated waters.

As it is visible from Fig. 11, while the pH is increasing to values from 9 to 10 it has experienced the highest Pb removal

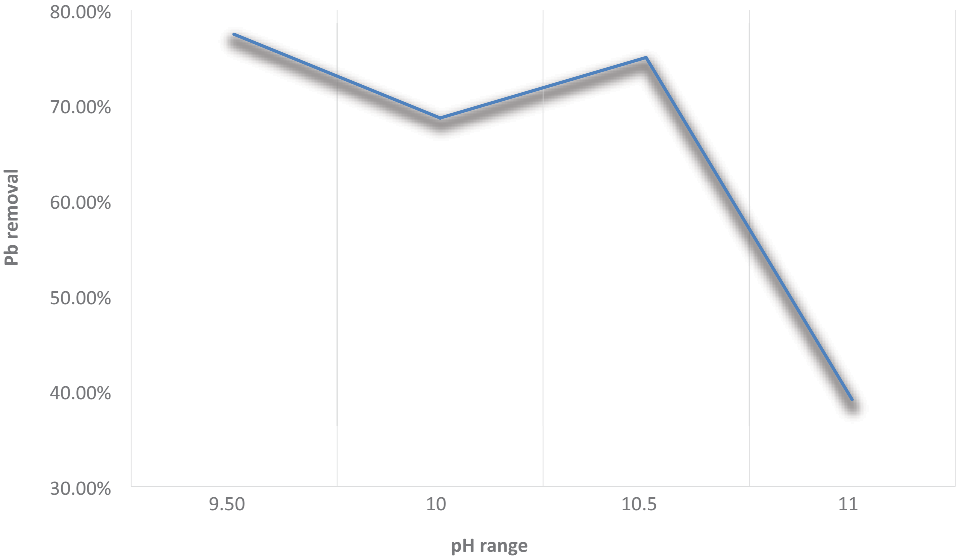

Fig. 13. Lead removal over different pH range values (site 2).

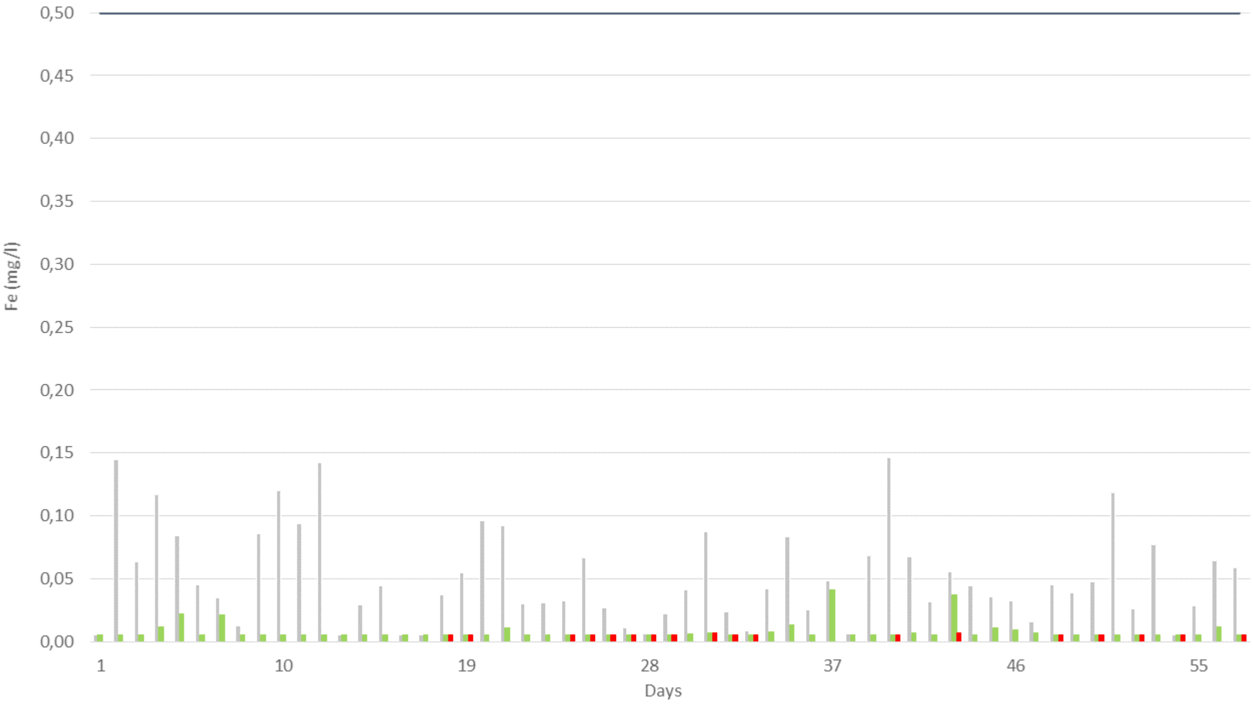

Fig. 14. Iron removal at Site 1. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, the blue line indicates the projects limit.

close to 70%. At pH values below 9 and above 10 the removal efficiency deteriorates significantly.

At Site 2 (Fig. 12) the infeed quality was even more challenging where the mean lead concentration was 86.26 ppb and the achieved effluent concentration following the filtration step was 25.27 ppb. The filtration removal efficiency was 70.69%. The contribution of RO system was even smaller by achieving a quality of water with 17.57 ppb and a removal efficiency of 30.45%.

The effect of the pH can be seen in Fig. 13. At pH 9–9.5 the highest removal was achieved (77.37%), while as the pH was increasing the removal rate was decreasing rapidly to rates below 40% at pH above 10.5.

The results are indicating that the removal of lead is hindered by increased pH. It is also obvious that the optimization of the filtration unit might render the RO system an impractical application for the specific water treatment procedures.

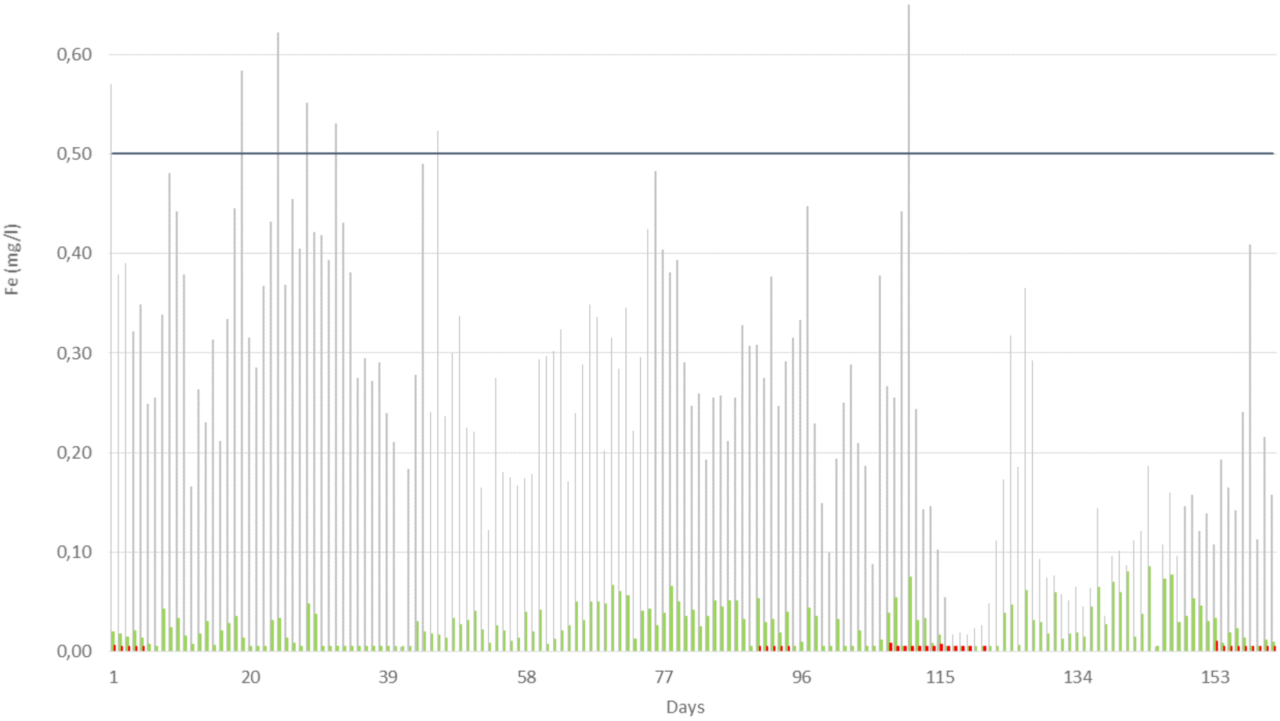

Fig. 15. Iron removal at Site 2. Grey lines represent the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the limit for project target.

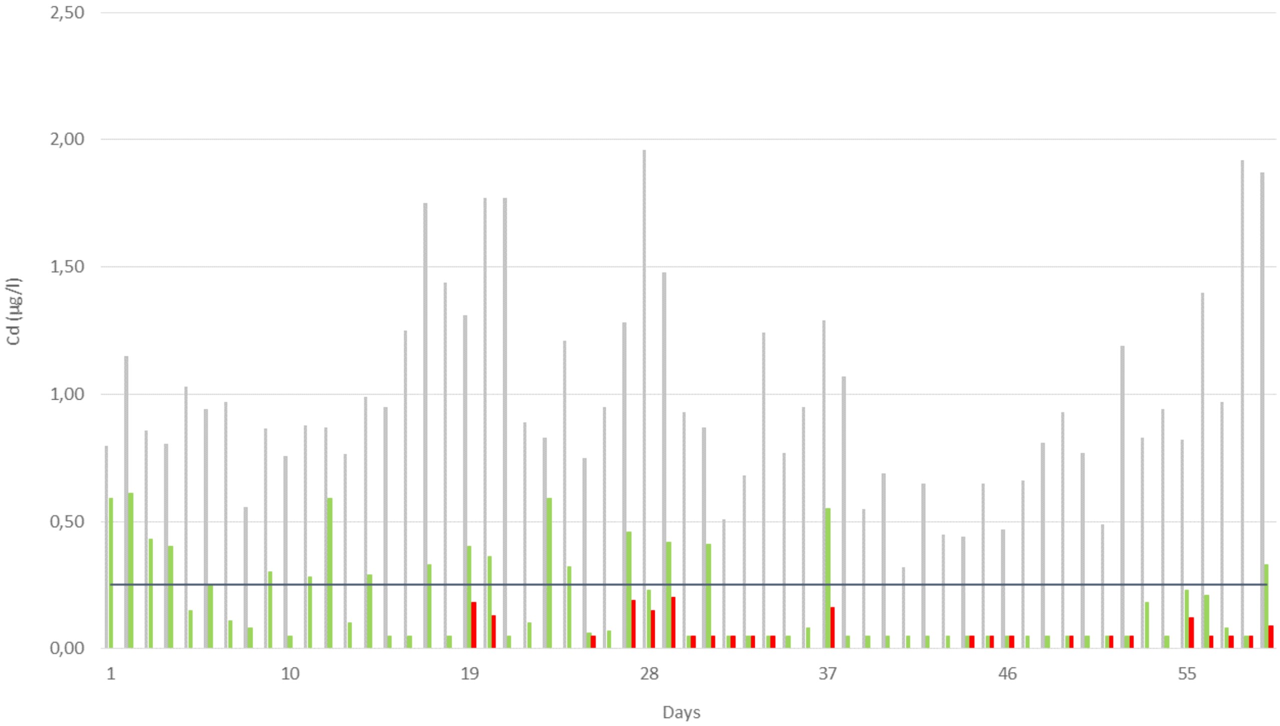

Fig. 16. Cadmium removal at Site 1. Grey line represents the concentration before the filtration, green lines after GAC, red lines after RO, and the blue line indicates the project target.

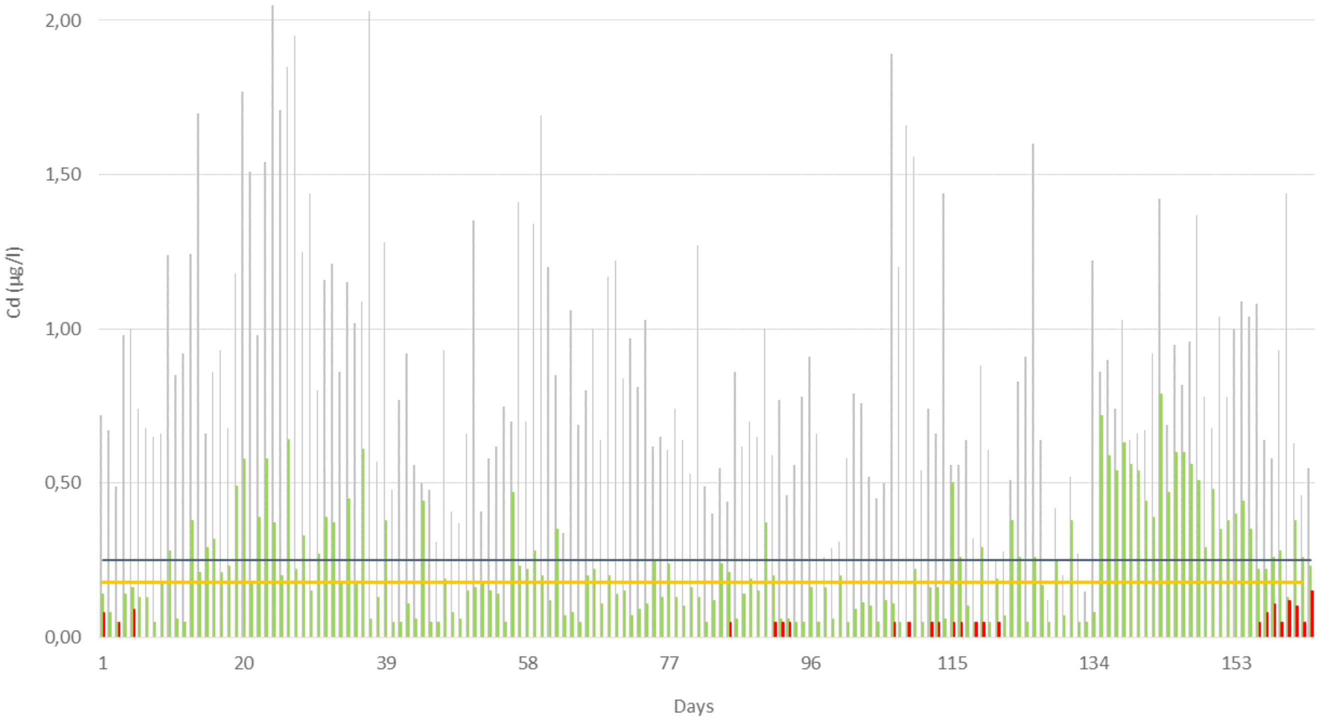

Fig. 17. Cadmium removal at Site 2. Grey line represents the concentration before the filtration, green line after GAC, red line after RO, the blue line indicates the project target, and the orange line mean annual concentration.

The iron removal was found very efficient with the suggested method (Fig. 14). The influent concentration is low, but the removal efficiency is high. The concentration in filtrated water is near to the limit of detection.

At Site 2 (Fig. 15), the influent was higher but still below the limit. The efficiency of the removal was similar as at Site 1, where the effluent concentration was close to the limit of detection.

The final metal that has been monitored, as far as it concerns the removal efficiency of the method, is cadmium (Cd^{2+}). At Site 1, the effluent concentration was below the limit for potable water. The annual average concentration should not exceed the 0.25 ppb and with the current method it was achieved an effluent of 0.08 ppb (Fig. 16).

At Site 2 (Fig. 17), the same result was exported. The cadmium concentration at the effluent was below the permissible limit for potable water.

Tables 1 and 2 show the removal efficiency in percentage of the PU compared with the mean heavy metal concentrations. The PU effluent met project limit targets which are near to potable water limits.

Based on the data retrieved from the pilot from two different sites, other parameters, except pH, should be evaluated for achieving a higher removal efficiency of lead and arsenic.

Table 1
Removal efficiency of metals at Site 1

Heavy metal	Mean effluent concentration	Mean concentration after filtration	Removal (%)	Project target (mean annual concentration)	Limit for potable water[a]
Pb (ppb)	27.2	10.9	59.8	7.2	10
Zn (ppm)	0.145	0.005	96.6	0.05	–
Fe (ppm)	0.042	0.005	88.0	0.5	0.2
Mn (ppm)	3.91	0.08	98.0	0.2	0.05
Cd (ppb)	0.89	0.08	91.0	0.25	5
As (ppb)	31.5	9.44	70.0	30	10

[a] European Directive 98/83/EC, and Greek Legislation JMD Y2/2600/2001, GG 630/B/26.4.2007, "Quality of water intended for human consumption [21,22]."

Table 2
Removal efficiency of metals at Site 2

Heavy metal	Mean effluent concentration	Mean concentration after filtration	Removal (%)	Project target (mean annual concentration)	Limit for potable water[a]
Pb (ppb)	88.5	25.6	71.0	7.2	10
Zn (ppm)	0.16	0.015	91.0	0.05	–
Fe (ppm)	0.25	0.021	91.6	0.5	0.2
Mn (ppm)	0.39	0.073	81.6	0.2	0.05
Cd (ppb)	0.770	0.18	76.6	0.25	5
As (ppb)	52.9	25.67	51.5	30	10

[a] European Directive 98/83/EC, and Greek Legislation JMD Y2/2600/2001, GG 630/B/26.4.2007, "Quality of water intended for human consumption [21, 22]."

The parameters that should be optimized are the flow rate and the NaOCl dosing.

4. Conclusions

The PU is based on the oxidation of heavy metals with modified CSFs and activated carbon filters. The process is very effective with proper pH and redox regulation and it has been demonstrated that it can effectively reduce the concentration of specific heavy metals below the permissible levels requested. The removal ratio for most of the contaminants is marginal and noncost effective when RO is applied as polishing stage. The pilot plant provided clear evidence that:

- the removal of Fe, Mn, and Zn is excellent and ranges at values from ~90% to ~100%.
- the removal of Cd is very good and ranges at values of 80%–90%.
- the most challenging metals were As and Pb, where the removal was below 80%. Further optimization required for achieving a higher removal on Pb. More recent data, that followed the optimization procedures, show a higher removal efficiency and effluent concentrations below the projects target. The data will be presented in upcoming works.

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