

The Removal of Heavy Metals from the Leachate of Aged Landfill: The Application of the Fenton Process and Nanosilica Absorbent

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ABSTRACT

Since leachate is typically composed of numerous constituents, its management requires special attention. After the raw leachate of Saravan in Rasht (Guilan Province, Iran) was transferred to a laboratory and its specifications were determined, it was subjected to experiments by the bench-scale method. The analyses of pH and heavy metals were performed in the main and control anaerobic reactors at time zero, before precipitation, and two hours after precipitation. After the anaerobic process was over and the optimal retention time was identified in the anaerobic reactor, the removal of heavy metals was analyzed by the Fenton process and nanosilica absorbent in leachate treatment. In the primary anaerobic reactor, the highest and lowest removal rates were 59 and 39% for Ni and Cu, respectively. In the Fenton process with optimal H_2O_2/Fe^{+2} ratio, Cu and Hg showed the lowest and highest removal rates of 22.4 and 54.54%, respectively. At the optimal rate of nanosilica absorbent and the retention time of 15 min, As was removed maximally with an efficiency of 38% and Cu was removed minimally. The results revealed that the integration of the anaerobic process with the Fenton process and nanosilica absorbent was very effective in removing heavy metals from the aged landfill leachate.

1. INTRODUCTION

The growth of industries and the development of technology over the past few decades have increased the production of solid waste (Sheng and Chih, 2000). There are various ways to dispose or use municipal waste including separation, hygienic disposal, incineration, and composting. Hygienic disposal is the simplest and least expensive method of waste disposal that is most commonly used in Iran and even the world. However, hygienic disposal has a major problem - the generation of leachate, which is very harmful to health and the environment (Ghasemi and Hagalifard, 2014). The Rasht city is one of the most important cities in the north of Iran. The Rasht area is 136 km² and generates about 500 tons of waste per day (Pirouz et al., 2010). The Saravan landfill is the largest dumpsite in the north of Iran. The Saravan landfill is located in the south of Rasht city, Iran (Karimpour-Fard et al., 2020).

Biological reactors are capable of decomposing or removing the compounds of waste to the extent that the toxic compounds of the waste leachate are reduced

to below the acceptable standards for drinking water or groundwater (Taghipour, 2009; Kiani et al., 2015).

After years in different cells and parts of the landfill, different phases of decomposition may be in progress. Leachate production is significantly reduced by replacing the final coating. In evaluating the long-term sustainability of a landfill, it should be considered that the coverage of the landfill will shrink. When the landfill coverage vanishes, the amount of leachate will increase even long after the landfill is closed (Taghipour, 2009).

The variations of the leachate compounds and the quantity of pollutants removed from the waste often depend on the age of a landfill expressed as the time of waste decomposition or the calculation of the time of the first leachate emergence. The landfill age obviously plays an important role in leachate characterization, which is a function of the type of waste stabilization processes. It should be emphasized that any changes in composition depend on the amount of water leaked into the landfill, too. Leachate contamination load generally maximizes in the first

years of landfill use and then, it decreases after several years. This trend generally applies to the main indices of pollution (TOC, BOD, and COD), microbial population, and major inorganic ions (heavy metals, Cl, and SO₄) (Saadatmand, 2012).

One of the most important parameters is the organic component of waste, which is biodegradable. Furthermore, the organic component of waste has a significant effect on landfill decomposition and therefore affects the quality of leachate. In the next step, the presence of substances that have a toxic or inhibitory effect on bacterial growth and disrupt the biodegradation process is of importance. Also, metals are released from the waste mass into the leachate in acidic conditions. When water seeps from decomposing wastes, both biological materials and chemicals penetrate the leachate. Since numerous components constitute the leachate, special attention should be paid to its management (Saadatmand, 2012).

Leachate treatment by Fenton can improve its quality, including odor, color, and organic matter content remarkably. Fenton is capable of greatly reducing toxic and resistant organic compounds and increasing biodegradable organic compounds. This reaction is mainly based on the generation of OH radicals by catalytic decomposition of H₂O₂ in an acidic medium. Common Fenton, electro-Fenton, and photo-Fenton processes have recently been evaluated for leachate treatment (Neyens and Baeyens, 2003).

Due to the uniform nature of its decomposition process, Fenton is a simple process and no energy is needed to activate the process, thus reducing its energy consumption. The disadvantages of the Fenton process are the high cost of its operation due to the need for chemicals and the cost of sludge disposal (Kiwi et al., 2000).

Hydrogen peroxide (H₂O₂) and iron ions (Fe²⁺) are the two major reaction agents in the Fenton process that produce hydroxyl radicals. Therefore, the concentration of H₂O₂ is an important factor in the oxidation of organic compounds and the progress of the oxidation process, and the ratio of H₂O₂ to organic matter that is being oxidized is an important parameter. The amount of H₂O₂ use is, on the other hand, an important economic factor in the Fenton reaction and is the main reason that the Fenton process is cheaper than other advanced oxidation processes (Wang et al., 2004).

Generally, Fenton oxidation consists of four steps: pH adjustment, oxidation, neutralization, and coagulation and precipitation. Since iron salt cannot be

preserved during the decomposition process, the Fenton process generates a large amount of fine coagula that contain iron hydroxide as the side product of the precipitation and should be removed from the system (Wu et al., 2004).

The removal mechanisms are the use of the coagulation-flocculation process for organic compounds, which mainly contain humic acids, the attachment of cationic metal species to some parts of it, thereby neutralizing humic substances and reducing their solubility, and the adsorption of humic substances on non-crystallized metal hydroxide precipitates (Yu et al., 2003). The combination of the Fenton oxidation with coagulation and flocculation can have a synergistic effect on the benefits of the treatment while overcoming their limitations. A limitation of this type of combined treatment is that it prefers an acidic medium for the decomposition of organic matter whereas, in the coagulation process that uses FeCl₃, the coagulant performs better at pH 4 to 6 (Cui et al., 2020).

2. METHODOLOGY

2.1 Anaerobic process

Raw leachate was transferred from Saravan, Rasht to a laboratory to determine its characteristics, and the assay was performed by the bench-scale method (Table 1). In Pilot 1 as the main anaerobic reactor, 2.5 L of the raw leachate was first poured into a glass container. Then, 3 g of Guigoz milk powder was added to provide the macro and microelements required for microbial reinforcement and growth in the pilot reactors, and 250 g of activated sludge (from Pegah Factory of Guilan) was added to the reactor as the nutrient (Figure 1). Analyses to determine pH and heavy metals, including Cu, As, Ni, Mn, and Hg, in the reactor containing leachate, seed, and nutrient were carried out at time zero, before precipitation, and two hours after precipitation (Eaton, 2005). The amount of heavy metals was estimated by Inductive Couple Plasma spectrometry (ICP-Qes-Spectro arcos, Germany). Instrument conditions were: ICP-QES (Spectro arcos); pump rate, 30 rpm; ICP torch injector, 2.5 mm; RF power, 1,400 w; plasma gas flow rate, 14.5/min; auxiliary gas flow rate, 0.9/min; and nebulizer gas flow rate, 0.85/min.

Five glass reactors were made with a volume of 3 L, and the same amounts of leachate, activated sludge, and milk powder were added to them. Then, the lid was closed to create anaerobic and batch conditions. Each reactor was placed on a mixer with

five different retention times of 5, 10, 15, 20, 25 days. After the retention time was over, the reactor mixer was switched off and after two hours of settlement, the pH and Cu, As, Ni, Mn, and Hg were analyzed.

The experiment was carried out in Pilot 2 of the anaerobic reactor as the control reactor with the same times specified in the previous reactor and the analyses described in the previous reactor without the addition of seed or nutrient (Kheradmand et al., 2009).

Table 1. Specifications of raw waste leachate of Saravan, Rasht

pH	8.2
Copper	0.313 mg/L
Arsenic	0.033 mg/L
Nickel	0.138 mg/L
Manganese	1.48 mg/L
Mercury	0.011 mg/L



Figure 1. The primary reactors with retention times of 5-25 days

2.2 Examination of the Fenton process in the main reactor

After the anaerobic phase was over and the optimal retention time was determined in the anaerobic reactor, the Fenton process was explored. The study used an H_2O_2 solution with a weight percent of 35% and a mass volume of 1.13 and iron sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$).

First, the leachate was poured into the container and its pH was adjusted to the desired level by sodium hydroxide and sulfuric acid (98% w/w). In the next step, Fe^{2+} was adjusted to the desired concentration by

adding iron sulfate and the optimal amount of Fe^{+2} was obtained. Then, a certain volume of H_2O_2 was added and after the reaction time was passed, the optimal amount of H_2O_2 was attained. Finally, the optimal molar ratio of $\text{H}_2\text{O}_2/\text{Fe}^{+2}$ was obtained (Figure 2). Finally, the result was given 30 min for the formed sludge to precipitate. The heavy metals Cu, As, Ni, Mn, and Hg in the supernatant were, then, measured for treatment efficiency. The jar test device was first set at 200 rpm for 30 min for rapid mixing and then at 120 rpm for 60 min for slow mixing (Kargi and Pamukoglu, 2003).



Figure 2. The study of the Fenton process in the jar test at different ratios of $\text{H}_2\text{O}_2/\text{Fe}^{+2}$

2.3 Efficiency of silica nanoparticles

To determine the efficiency of nanosilica absorbent in the treatment of waste leachate, the efficiency of the removal of heavy metals Cu, As, Ni, Mn, and Hg was estimated at all steps for retention times of 15-75 min as per the standard guideline (Kashitarash et al., 2012). Nanosilica is extensively used in the industry. Due to its higher specific surface area, nanosilica has higher absorbance potential than the micrometer state at the nanoscale (Tzvetkova and Nickolov, 2012). Experimental nanosilica adsorbent properties were: non-crystalline, 99.5% purity, 20-30 nm, and specific surface area 180-600 m²/gr.

3. RESULTS AND DISCUSSION

3.1 Removal of heavy metals by the anaerobic process

The efficiency of the anaerobic reactor in removing heavy metals was investigated. According to Figures 3 and 4, the optimal retention time for heavy metal removal was 20 days in the primary and control reactors. In the primary anaerobic reactor (Figure 3), the highest removal rate was 59 and 39% for Ni and Cu and in the control reactor (Figure 4), the lowest removal rate was 48, 12, and 12% for As, Mn, and Hg, respectively. In present study, approximately, the removal percentage of all metals under aerobic bioreactor was higher than the control bioreactor.

Kheradmand et al. (2009) emphasized biological methods for the removal of heavy metals from wastes due to their advantages as they are economical and environmentally friendly. They measured the removal rate for six metals of Cu, Fe, Mg, Mn, Ni, and Zn. The anaerobic reactors showed a higher capability in removing heavy metals as they

generated adequate sulfide for sequencing heavy metals. The removal rates of Cu, Fe, Mg, Mn, Ni, and Cu per unit of input at the optimal load, i.e., 2.2 g/L, were 100, 88, 0, 100, 82, and 36% in the first anaerobic digester and 15, 0, 67, 37 and 25% in the second anaerobic digester, respectively (Fouladifard et al., 2008). Qiu et al. (2016) reported that the removal efficiency of Zn, Cd, Ni, and Cr was 89.8, 100, 52 and 31.1%, respectively. High heavy metal concentrations inhibit the anaerobic co-digestion process, resulting in reduction of removal of organic substances and biogas (Nguyen et al., 2019). In a study by Kalyuzhnyi et al. (2003), the removal efficiency of Cd, Pb, Cu, Zn, and Fe was perfect with three anaerobic methods used by concomitant sequencing in the form of sulfides that were insoluble in the sludge bed. According to Bilgili et al. (2007), metals started to precipitate after reaching the methanogenesis phase and the increase in pH up to the neutral level. Our results are consistent with Kheradmand et al. (2010) and Qiu et al. (2016). The different removal rates of heavy metals at the retention time of 20 days is likely to be related to the capability of anaerobic bacteria including Sulfate-reducing bacteria and cyanobacteria in biologically converting some metal ions into sulfides (Lefebvre et al., 2007).

3.2 Data derived from the Fenton process

Based on the results concerning the effect of the Fenton process on the removal of As, Cu, Hg, Mn, and Ni from the leachate of the Saravan landfill, the highest and lowest removal rate at the optimal ratio of H₂O₂/Fe⁺² were 22.4 and 54.54% related to Cu and Hg, respectively (Table 2).

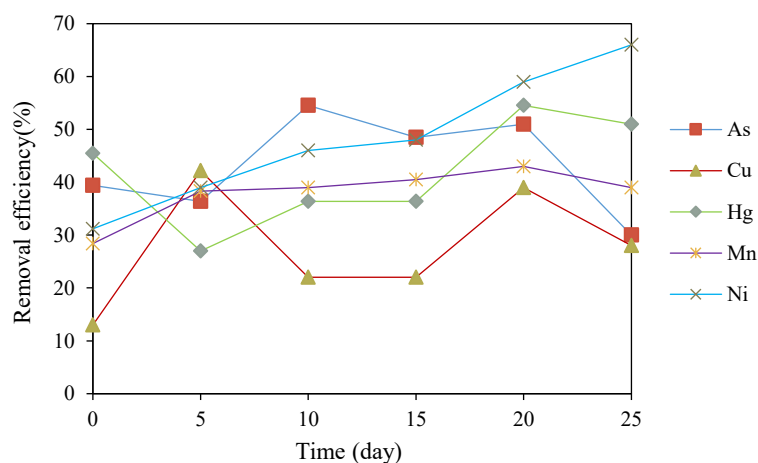


Figure 3. The removal efficiency of heavy metals As, Cu, Hg, Mn, and Ni in the primary reactor with the retention time of 5-25 days

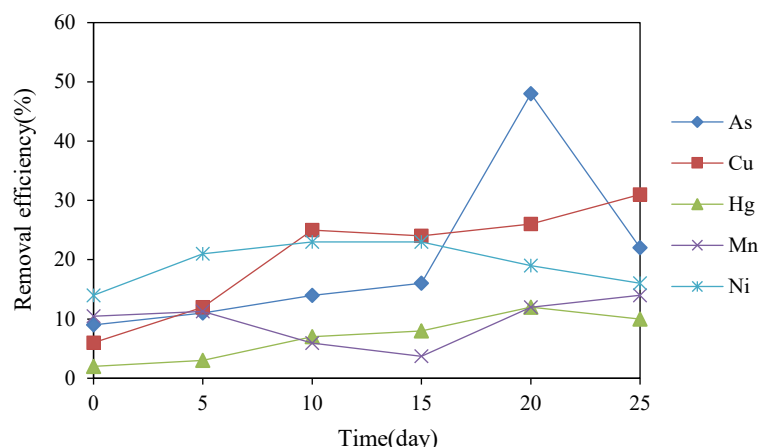


Figure 4. The removal efficiency of heavy metals As, Cu, Hg, Mn, and Ni in the control reactor with the retention time of 5-25 days

To study the effect of iron ion concentration on this process, [Malakootian et al. \(2011\)](#) set it at 100, 200, 400, 800, 1,600, and 3,200 mg/L. The highest Cr removal rate of 99.7%, COD of 68 %, and turbidity of 97.6 % were obtained from Fe concentrations of 1,600, 800, and 400 mg/L, respectively. An increase in Fe ion concentrations beyond these levels reduced the efficiency of Cr removal, COD, and turbidity, which may be attributed to the tendency of hydroxyl radicals to oxidative-reductive reaction with Fe^{+2} and H_2O_2 . In our experiment, BOD and COD efficiency in the Fenton process was found 95.9 and 75% at Fe^{2+} rate of 1,800 mg/L and 95.3 and 83.3% at H_2O_2 rate of 4,500 mg/L, respectively. [Zazouli et al. \(2012\)](#) evaluated the removal of Fe, Cu, and Cr. In general, since Fe was added to all processes as a catalyzer, it was increased in both effluents and the generated sludge, which was a constraint of the Fenton-based process. The application of UV radiation reduced the Fe content of both sludge and effluent. As well, the Cu removal rate reached over 70% in the Fenton and photo-Fenton processes. The lowest removal rate of Cu was about 28% in the modified Fenton process. The removal rate of Cr was 100% in the photo-Fenton process. In a study reported by [Malakootian et al. \(2010\)](#), the maximum Ni removal rate was 98 % obtained under the optimal conditions, the contact time of 60 minutes, the pH of 4, the Fe^{+2} content of 1,600 mg/L, and the H_2O_2 content of 2,500 mg/L. [Azhdarpoor et al. \(2015\)](#) reported that when the Fenton reaction was applied in the biological sludge, the removal rate reached 75.3, 72.6, 34.5, and 65.4% for Zn, Cu, Pb, and Cd, respectively. According to [Malakootian et al. \(2011\)](#), the removal of heavy metals including Cr, organic matter, and turbidity by the Fenton process is affected by diverse factors such as oxidant concentration,

catalyst, contaminant concentration, pH, and reaction time. These factors played a significant role in the generation of hydroxyl radical and the efficiency of the Fenton process so that higher H_2O_2 content caused the floatation of sludge and the disruption of biological purification after the Fenton process, higher Ferro-iron content increased TDS and EC of the effluent making it necessary to treat the generated sludge, and higher pH beyond the optimal level reduced the generation of hydroxyl radicals, the rapid decomposition of H_2O_2 into water and oxygen, and the precipitation of Ferro-iron with longer contact time resulting in higher treatment costs. Our results are in agreement with [Azhdarpoor et al. \(2015\)](#).

Table 2. The removal rate of heavy metals with the optimal $\text{H}_2\text{O}_2/\text{Fe}^{+2}$ ratio

Metals	Mn	Ni	As	Cu	Hg
Removal rate (mg/L)	46	32	51	22.4	54.54

3.3 Data derived from nanosilica absorbent

According to the results concerning the effect of the optimal amount of nanosilica absorbent on the removal of As, Cu, Hg, Mn, and Ni from the leachate of the Saravan landfill, the highest removal efficiency at the retention time of 15 min was 38 and 25% for As and Cu, respectively and it was 58 and 31.25% for Hg and As at the pH of 3, respectively ([Figure 5](#)). When the retention time was increased to 30 min, Mn and Ni removal rates were increased slightly, but further increase in the retention time to 75 min resulted in the reduction of their removal efficiencies. At the nanosilica absorbent rate of 0.5 g/L, the retention time of 15 min, and the pH of 9, the highest and lowest removal rates were 97.36% and 10% related to Hg and Cu, respectively.

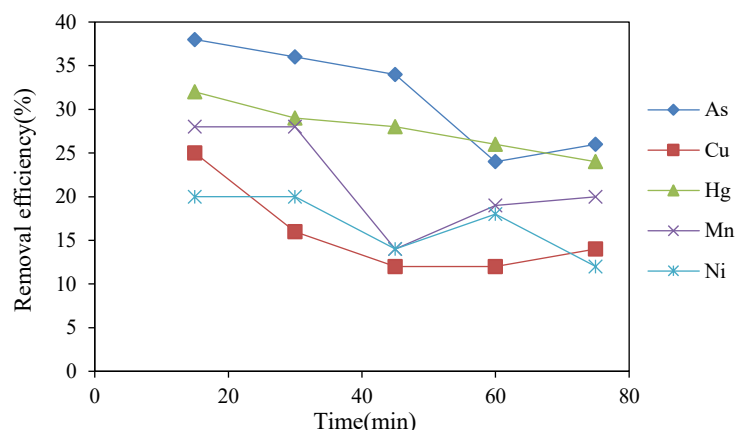


Figure 5. The removal efficiency of heavy metals As, Cu, Hg, Mn, and Ni at different retention times with 4 g/L nanosilica

In [Onyeji and Aboje's \(2011\)](#) study, 2 g of activated carbon entailed over 80% removal of Hg(II) and Pb(II). As well, the absorption of Hg(II), Pb(II), and Cu(II) by the activated carbon depended on the absorbent amount and initial metal concentrations. [Onundi et al. \(2011\)](#) reported that under laboratory conditions, nano-size composite resulted in the optimal absorption of metals at a pH of 5, an amount of 1 g/L, and a contact time of 60 min. [Kiani et al. \(2015\)](#) found that the application of all five coagulants reduced the concentration of residual heavy metals below the standard limits of treated effluents of Iran. The efficiency by which poly-ferric sulfate removed heavy metals and COD from the leachate with a pH of 11 reached 70-87 and 50%, respectively ([Figure 6](#)). [Mojiri et al. \(2015\)](#) studied three SBR reactors with 3 g/L of powdered ZELIAC, powdered activated carbon, and powdered zeolite with 90 min of settling time and 20% of leachate-to-wastewater mixing ratio. The reactor containing powdered ZELIAC exhibited an efficiency of 79.24% for Cr removal and outperformed the other reactors. Zeolites are naturally occurring silicate minerals

whose capability of cation exchange is a decisive factor for the removal of heavy metals from industrial sewage ([Hlihor and Gavrilescu, 2009](#)).

[Kocaoba et al. \(2007\)](#) carried out several trials on the removal of heavy metals from aqueous solutions using clinoptilolite in Biga-Canakkale, Turkey. They determined the efficiency of zeolite absorbent in removing Cu(II), Cd(II), and Ni(II) from the aqueous solutions at different initial concentrations, zeolite rates, agitation speeds, and pHs. The best metals selected in this study were Cd(II)>Ni(II)>Cu(II). The rate of metal absorption to zeolite showed that the process was fast and the maximum absorption happened at the first contact time. This very slow initial absorption was subsequently stabilized and saturated in 20-30 min. [Johnson et al. \(2008\)](#) reported that chemically enhanced primary treatment with 40 mg/L of ferric chloride and 0.5 mg/L of polymer yielded over 200% efficiency in removing Cr, Cu, Zn, and Ni and it was 47.5% for Pb removal as compared to traditional primary treatment. Our results are consistent with [Kiani et al. \(2015\)](#).

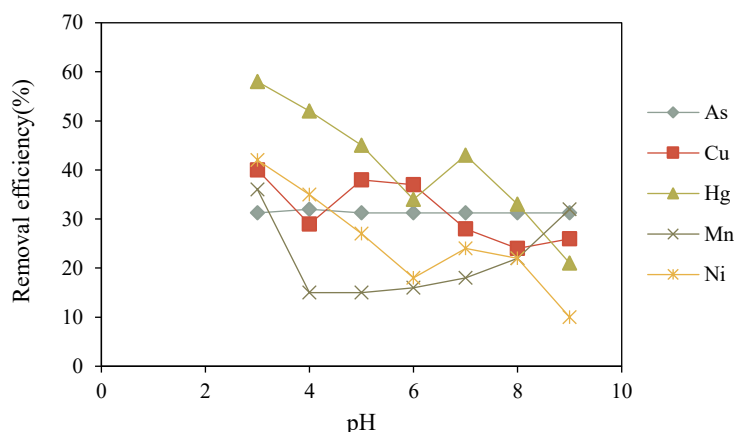


Figure 6. The removal efficiency of heavy metals As, Cu, Hg, Mn, and Ni at different pH values with 4 g/L nanosilica

Increasing coagulator dosage beyond the optimal level results in the re-stabilization of colloids (Ayeche, 2012). We found that the integration of the anaerobic process with the Fenton process and nanosilica absorbent was very effective in removing heavy metals from aged leachate (Figure 7). Feki et al. (2020) found that batch and semi-continuous

anaerobic fermentations had a positive effect on the electro-Fenton (EF) pretreatment in enhancing the biogas potential and stability of the anaerobic system. They revealed that the EF process can be a more consistent solution for the improvement of waste-activated sludge anaerobic treatment.

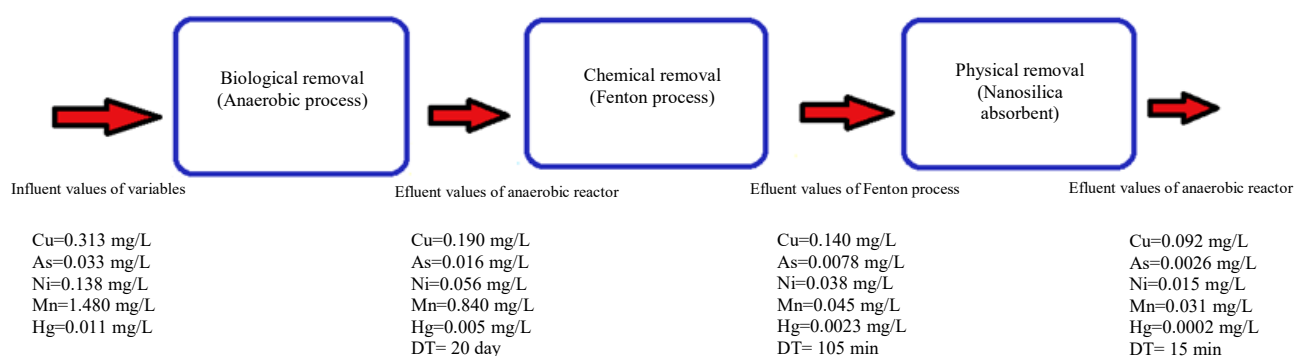


Figure 7. Steps of biological, chemical and physical removal of leachate in the primary reactor

4. CONCLUSION

The integration of the anaerobic process with the Fenton process and nanosilica absorbent was very effective in removing heavy metals from aged leachate. Since Fe was added to all processes as a catalyst, it was increased in both effluents and the generated sludge and this was a constraint of the Fenton-based process. Regarding the effect of the Fenton process on the removal of some heavy metals including As, Cu, Hg, Mn, and Ni from the experimental leachate, the utmost and minimum removal rate at the optimal ratio of $\text{H}_2\text{O}_2/\text{Fe}^{+2}$ were 22.4 and 54.54% related to Cu and Hg, respectively. The optimal retention time for heavy metal removal was 20 days in the primary and control reactors. Since old leachates have a lot of non-biodegradable organic matter, anaerobic treatment should be used in the first stage to remove biodegradable organic matter, and in the next steps the Fenton and nanosilica adsorbent process removes non-biodegradable organic matter. Further studies are suggested to perform on the nanosorbents with higher adsorption capacity, such as carbon nanocomposites, on old leachate as well as on young leachate.

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