

Efficiency of Washing Fluids for Reducing Toxic Metals from ETP Sludge

Evan Johnson¹, Marielia Young², Davis Ross³

Associate Professor¹, Pro Vice Chancellor², Professor³

Department of Civil Engineering

Imperial College of Engineering

Corresponding Authors' Emails: young_marielia89@yahoo.co.in

Abstract

The indiscriminate disposal of Effluent Treatment Plant (ETP) sludge from industries leads to soil, groundwater and surface water pollutions. To avoid the high costs associated with the disposal of large volume of this sludge, this material can be used in some building materials or can be used as fertilizer. As the accumulation of heavy metals in plants causes threat to living beings, it has to be pretreated to remove heavy metals from the sludge to required levels. Soil washing can be used to separate heavy metals from the sludge. For the present study, ETP sludge was assessed to know the removal efficiencies of five leaching solutions. The leaching solutions attempted were distilled water, 0.1 N HCl, 0.1 N EDTA, 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl₃. Column leach tests were conducted on the ETP sludge using the selected leaching solutions and the migration rates of different metal ions were determined. The efficiencies of different leaching solutions were compared and was found that 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl₃ are the most suitable solutions to remove heavy metals from this sludge.

Keywords: HCl, EDTA, FeCl₃, Dispersion coefficient, Retardation factor

INTRODUCTION

The highly contaminated sites are generally found at industrial areas which often contain metals such as Lead,

Chromium, Cadmium, Nickel and Zinc (Gusiatin and Klimiuk, 2012). Unlike organic contaminants most of the heavy metals do not undergo microbial or

chemical degradation (Kirpichtchikova et al., 2006). The heavy metal contaminated sites cause threats and hazards to humans and the ecosystem in the following ways (McLaughlin et al., 2000a; McLaughlin et al., 2000b; Ling et al., 2007; Wuana and Okieimen, 2011).

- i. By direct contact with contaminated soil
- ii. Through the food chain (soil-plant-human)
- iii. By contaminating the ground and surface water resources
- iv. Reduction in agricultural land

The treatment of industrial effluents as part of pollution control technologies results in the generation of large volume of solid phase pollutants (sludge). The sludge that retained at the effluent treatment plants (ETP) after the solids separation process is called ETP sludge which creates a lot of environmental problems due to lack of disposal methods (Rajkumar and Hema, 2011). The indiscriminate disposal of this ETP Sludge leads to soil, groundwater and surface water pollutions. Due to the seriousness of this, the ETP Sludge was listed as hazardous waste in India by Ministry of Environment and Forests (MoEF, 2008) and landfill disposal was recommended for inorganic sludge. As the costs associated with transport of

this large volume of sludge and construction of landfills are very high, recycling this waste as substitute for building materials (like bricks and low cost concrete) is a better option (Balasubramanian et al., 2006; Baskar et al., 2006; Mary Lissy and Sreeja, 2014; Shivanath et al., 2015). Kakatiet al., 2013 have studied the use of Textile ETP sludge as a fertilizer at low concentration to enhance the plant growth. They found that 10% sludge plus 90% farmyard enhances the plant growth and stated that the accumulation of toxic materials in the plant system after the treatment should be studied in detail. Hence we may use the ETP sludge as fertilizer if the pollutants are removed to the required levels by using soil washing technique.

Soil washing may be in-situ or ex-situ which involves addition of chemicals to water to dissolve the metals. The commonly used flushing solutions are Plain water, Surfactants and/or co-solvents, Acids, Bases, Chelates and Solvents (Diane, 1997).

Gebreyesus, (2014) have discussed in detail about the research on remediation techniques and concluded that chemical extractants are the most cost effective and less damaging, including organic acids and

chelating agents. Mulligan et al., (1999a) and Mulligan et al., (1999c) indicated that biosurfactants, biologically produced surfactants, enhance the removal of metals from contaminated soils and sediments.

Uwumarongie-Ilori and Okieimen, (2010) also used the soil washing technique to treat Chromated Copper Arsenate (CCA) contaminated soil obtained from a wood treatment factory site by four organic acids i.e., oxalic, malonic, succinic and citric acids.

The commonly used chelating agents for soil washing include Ethylene diaminetetraacetic acid (EDTA), Nitrilotriacetic acid (NTA), Diethylenetriaminepentaacetic acid (DTPA), Ethylenediaminedisuccinic acid (EDDS) and citric acid (CA) which form stable complexes with most heavy metals over a broad pH range (Oustan et al., 2011 and Bilgin and Tulun, 2015). EDTA has the ability to chelate with almost all heavy metals (Zhou et al., 2011). Naghipoor Khalkhaliani et al., (2006) have used various concentrations of EDTA for evaluating the soil washing technique on an artificially contaminated sandy loam soil.

Oustan et al., (2011) used two natural, low molecular weight organic acids, oxalic acid (OA) and acetic acid (AA) to decontaminate a highly contaminated calcareous soil. Ma et al., (2014) have used 0.01 N FeCl₃ in thermal treatment of Mercury contaminated soil. Bilgin and Tulun, (2015) have also used 0.01 N FeCl₃ to treat coarse grained and fine grained soils which showed higher removal of zinc in the presence of iron. Akahane et al., (2013) and Hirokazu et al., (2010) have used FeCl₃ to treat Cadmium contaminated soils. The ferric salts have the capability to adsorb metal ions (both cations and anions) by co-precipitation. The precipitated ferric hydroxide facilitates incorporation of heavy metal ions directly into the precipitate matrix in place of the similarly sized and charged ferric cations (Patoczka et al., 1998).

For the present study, the ETP sludge was collected from KIADB Industrial area, Doddaballapur, Bangalore. Batch leach tests were conducted to know the different pollutants present in the sludge and their levels. The metal ions present in the sludge are given in Table 1. The effect of washing sludge with different leaching solutions was studied to find the appropriate leaching solution to remove the metal ions. The leaching solutions attempted were

distilled water, 0.1 N HCl, 0.1 N EDTA, 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl₃. The removal rates and the number of pore volumes required to reduce different metal ions to the desired levels have been obtained through estimation of migration rates of these ions which can be established through detailed column leach tests. The transport parameters were obtained by matching theoretical elution

curves (with a set of assumed diffusion coefficients and retardation factors) with experimental elution curves.

ETP sludge was collected at a filter press, KIADB industrial area, Doddaballapur, Bangalore, India. The wet sludge was air dried and then sieved through 2mm sieve to determine the index properties which are given in Table 2.

Table 1. Chemical properties of ETP sludge

Pollutant	Quantity of metals in ETP Sludge (mg/kg)
Copper (Cu)	92.3
Zinc (Zn)	152.7
Iron (Fe)	414.5
Chromium (Cr)	74.2
Cadmium (Cd)	4.7
Nickel (Ni)	52.3
Lead (Pb)	11.2

MATERIALS AND METHODS

ETP Sludge

Table 2. Characteristics of ETP Sludge

Specific Gravity (G)	Liquid Limit (%)	Plastic Limit (%)	Shrinkage Limit (%)	Plasticity Index (Ip)	Maximum dry density (g/cc)	Optimum water content (%)
1.7	38%	Non-plastic	9.8	-	0.98	33.5

Batch Leach Test

The total mass of zinc (M_0) which is the sum of adsorbed mass (m_s) and pore fluid mass (m_0) presented in each sample was found from batch leach test. The concentrations of contaminants were found to be negligible when conducted the normal batch leach test with distilled water. Hence a modified method of batch leach test was used to find the concentrations of different metals in the soil. 20 gm of dry soil sample was mixed with 100 ml of 0.1N HCl + 0.1N EDTA. The mixture was kept on shaking machine for about 24 hours and then heated it upto boiling point to dissolve the crystalline forms of metals present in the soil. The solution was then centrifuged and supernatant solution was collected. The supernatant solution was filtered through a 0.45- μ m pore size membrane filter. The concentrations of metals present in the effluent solution were found by AAS and their quantities in the soil were estimated.

Column Leach Test

The column leach tests were conducted on these samples to know the removal efficiencies of four leaching solutions i.e., 0.1N HCl, 0.1 N EDTA, 0.1N HCl+0.1N EDTA and 0.1 N $FeCl_3$. The solution was allowed to pass through the soil sample and effluent samples were collected

periodically. The volume of effluent and amount of metals leached out (Δm) in each time increment were found. The number of pore volumes of flow (T_1) was calculated for each time interval. The ratio of increment of solute mass (Δm) to initial solute mass (M_0) is called Leaching Mass Ratio (LMR) and sum of these ratios is called Cumulative Leaching Mass Ratio (LMR_m). The LMR_m values were calculated corresponding to each time increment and plotted against the corresponding leached pore volumes (T_1) to get the experimental elution curves (i.e., breakthrough curves for leaching) (Shackelford and Michael, 1997). The apparatus used for column leach tests is shown in Fig 1.



Fig.1. Photograph of the experimental setup

MODELLING COLUMN LEACH TESTS

The Advective-Dispersive-Reactive Equation (ADRE) for the case advective-dispersive transport during leaching is as given below (Eq. 1) (Brusseau, 1996).

$$R \frac{\partial C_r}{\partial t} = D \frac{\partial^2 C_r}{\partial z^2} - v_s \frac{\partial C_r}{\partial z} \dots\dots\dots(\text{Eq. 1})$$

Where C_r is resident or pore fluid solute concentration within the porous medium Addiscott and Wagenet, (1985) explained the different numerical and analytical soil leaching models and modeling

$$\text{LMR}_m = \frac{\sum \Delta m}{M_0} = \frac{T^I}{R} - \frac{1}{2} \left\{ \left(\frac{T^I}{R} - 1 \right) \text{erfc} \left(\frac{R - T^I}{2 \sqrt{\frac{T^I R}{P_L}}} \right) + \left(\frac{T^I}{R} + 1 \right) \exp \left(-\frac{P_L}{R} \right) \text{erfc} \left(\frac{R + T^I}{2 \sqrt{\frac{T^I R}{P_L}}} \right) \right\} \dots\dots\dots(\text{Eq. 2})$$

LMR_m = cumulative Leaching Mass Ratio
= $\sum (\Delta m / M_0)$

Δm = mass of contaminant leached in a particular duration

M_0 = total mass of contaminant present in the soil

T^I = Number of pore volumes; R = Retardation factor;

P_L = column Peclet Number = $V_s L / D$;

A MATLAB program was prepared for the above equation and theoretical LMR_m values were found for trial values of D and R , and experimental values of T^I and P_L .

approaches. Shackelford and Michael, (1997) have explained the Analytical modelling of Column leach tests based on Leaching Mass Ratio approach. They conducted the column leach tests on a soil fly-ash mixture and the leaching of metal ions Cadmium, Lead and Zinc was evaluated.

The governing equation (based on Eq. 1) for modelling column leach test for the Advective-Dispersive leaching as suggested by Shackelford and Michael, 1997 is given below (Eq 2).

The theoretical elution curves were plotted and were matched with the experimental elution curves to find the transport parameters.

COLUMN LEACH TESTS ON ETP SLUDGE

The leaching solutions used were distilled water, 0.1 N HCl, 0.1 N EDTA, 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl_3 . The samples were compacted to a density of 0.98 g/cc (MDD) and a water content of 33.5% (OMC). The void ratio and porosity

of the test samples were 0.82 and 0.45 respectively.

The pore volume of the samples was 56.55 cm³ and average seepage velocities with a hydraulic gradient of 10 were 1.3×10^{-5} , 5.2×10^{-5} , 5.5×10^{-4} , 1.4×10^{-4} and 1.2×10^{-4} cm/s with distilled water, 0.1 N HCl, 0.1 N EDTA, 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl₃ respectively. During the column leach test, the seepage velocity increased with time due to the removal of contaminants and hence an average value

of seepage velocity is taken for the analysis.

The effluent samples collected at different time periods were analyzed with AAS to find the leached quantities of various metals present in the sludge. The cumulative Leaching Mass Ratios (LMR_m) were calculated corresponding to each time period. The experimental elution curves (number of pore volumes vs LMR_m) of the metal ions with different leaching solutions are given in Figs 2 – 6.

LEACHING OF DIFFERENT METAL IONS WITH DISTILLED WATER

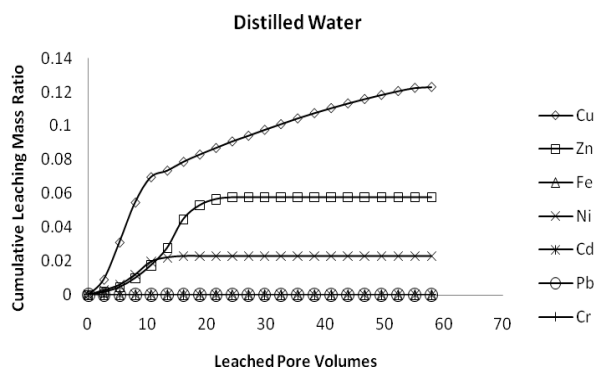


Fig.2. Experimental elution curves with Distilled water

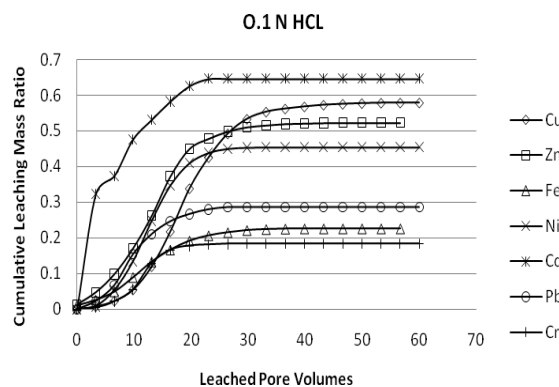


Fig.3. Experimental elution curves with 0.1 N HCl

As most of the metals are soluble in HCL, it has been used as leaching solution to remove the pollutants from the sludge. From the Fig 3, it can be observed that all the seven metals were leached out with this solution in different quantities. The metals Cd, Cu, Zn and Ni have almost reached a removal efficiency of 50% after passing around 25 pore volumes of the solution through the sludge. It can also be observed that after 25 pore volumes of flow, the leachability of metals was reduced and the elution curves were almost flat for all the metal ions tested.

The Order of removal efficiencies achieved with 0.1 N HCl is as given below.

Cd (64.5%) > Cu (57.9%) > Zn (52.3%) > Ni (45.3%) > Pb (28.5%) > Fe (22.6%) > Cr (18.3%)

LEACHING OF DIFFERENT METAL IONS WITH 0.1 N EDTA

As most metal ions form stable complexes with EDTA, its efficiency in removing pollutants from ETP sludge was studied by passing 0.1 N EDTA through this sludge. It can be observed from the elution curves of different metals (Fig 4) the curves were steep up to 20 pore volumes and were with

a moderate slope up to 30 pore volumes followed by a flat portion.

The metal ion Cd has reached its equilibrium much earlier compared to other metals at a pore volume of 10. The metal ions Cd, Cu and Zn have attained a removal efficiency of 80% and other metals have attained almost 50%. Hence this solution can be used to reduce the levels of contaminants in the sludge. The Order of removal efficiencies achieved with 0.1 N EDTA is as given below.

Cd (82.9%) > Cu (82.5%) > Zn (79.6%) > Ni (59.3%) > Pb (55.5%) > Fe (49.3%) > Cr (42.7%)

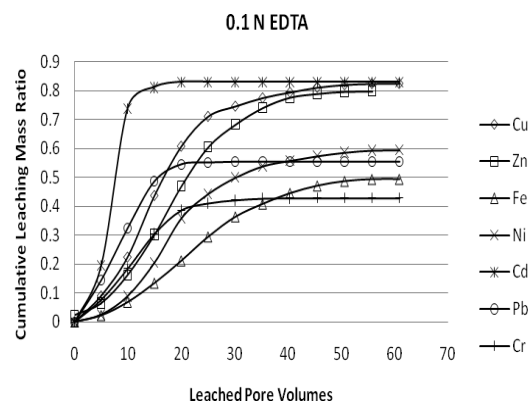


Fig.4. Experimental elution curves with 0.1 N EDTA

LEACHING OF DIFFERENT METAL IONS WITH 0.1 N HCl + 0.1 N EDTA

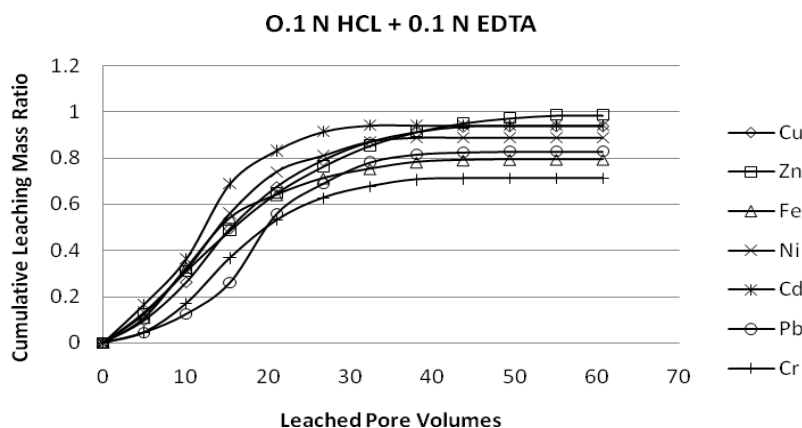


Fig.5. Experimental elution curves with 0.1 N HCl + 0.1 N EDTA

The sludge was also tested with 0.1 N HCl + 0.1 N EDTA to get the benefits of both acid and chelate. From the Fig 5, it can be observed that the elution curves of all the metals have followed the same trend indicating the effectiveness of this solution to remove the contaminants. The elution curves reached their equilibrium between 35-40 pore volumes of leaching with efficiencies of 70-95%.

The removal efficiencies achieved by this solution were much higher compared to HCl and EDTA alone (Figs. 3 and 4). The number of pore volumes required to achieve a particular efficiency were also less compared to those with previous solutions. The Order of removal efficiencies achieved with 0.1 N HCl + 0.1 N EDTA is as given below.

Zn (98.4%) > Cd (94.2%) > Cu (93.7%) > Pb (82.8%) > Ni (88.7%) > Fe (79.3%) > Cr (71.2%)

LEACHING OF DIFFERENT METAL IONS WITH 0.1 N FeCl₃

In the present study, the ETP sludge was also treated with FeCl₃ to know the removal efficiencies of different metals from this sludge. The experimental elution curves of different metal ions are as shown in Fig 6. It can be observed from these curves that the removal efficiencies were much higher with this solution compared to all the leaching solutions used. The Cd and Pb have reached their equilibrium much earlier at 15 pore volume of leaching with this solution compared to that of 0.1 N HCl + 0.1 N EDTA where the equilibrium was attained at 25 pore

volumes for these metals. But for the remaining metals the number of pore volumes required to achieve a particular removal efficiency were higher with this solution compared to those with 0.1 N HCl + 0.1 N EDTA. The order of removal capacities for removing different ions using this leaching solution is as given below.

Cd (98.8%)>Pb (98.7%) >Zn (97.2%)>Cu (95.8%)>Fe (82.8%)>Ni (79.9%)>Cr (72.1%) *See Figure: 6*

THEORETICAL ELUTION CURVES

Theoretical elution curves have been developed based on Eq. 2 using MATLAB v7 software tool to determine the transport parameters of different metal ions. As the Ferric Chloride and HCl + EDTA solutions have given comparatively best results, the transport parameters were determined with respect to these solutions. The theoretical elution curves obtained are shown in **Figs 7 – 20** for the leaching solutions. The transport parameters obtained are given in **Table 3**

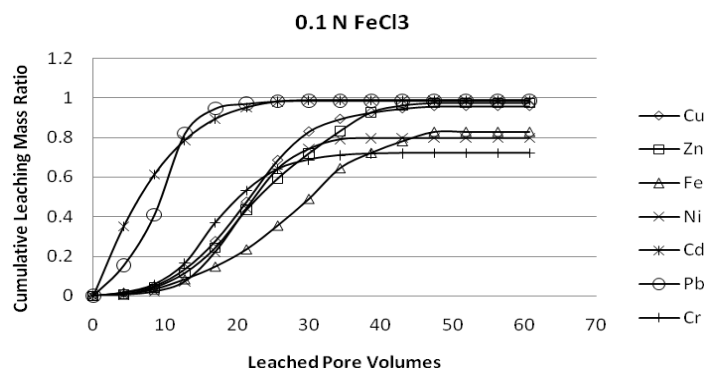


Fig.6. Experimental elution curves of different metal ions with 0.1 N FeCl₃

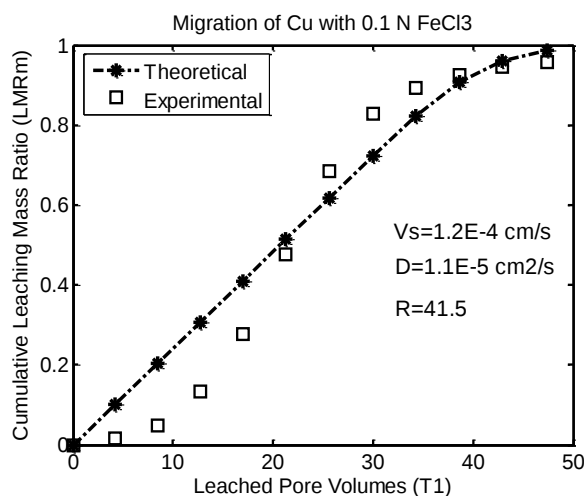


Fig.7 Theoretical elution curve of Copper with 0.1 N FeCl₃

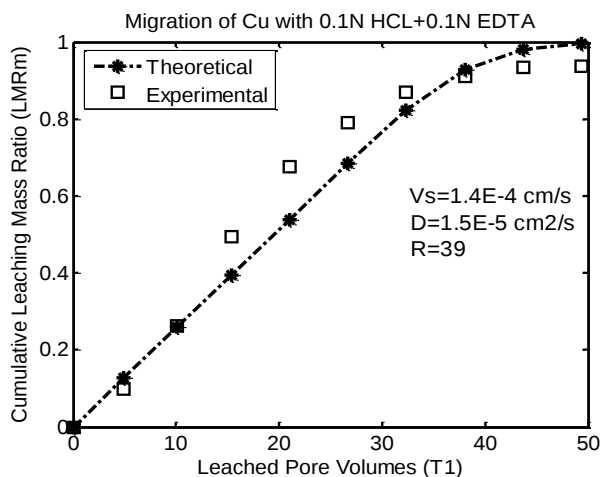


Fig.8 Theoretical elution curve of Copper with 0.1 N HCl + 0.1 N EDTA

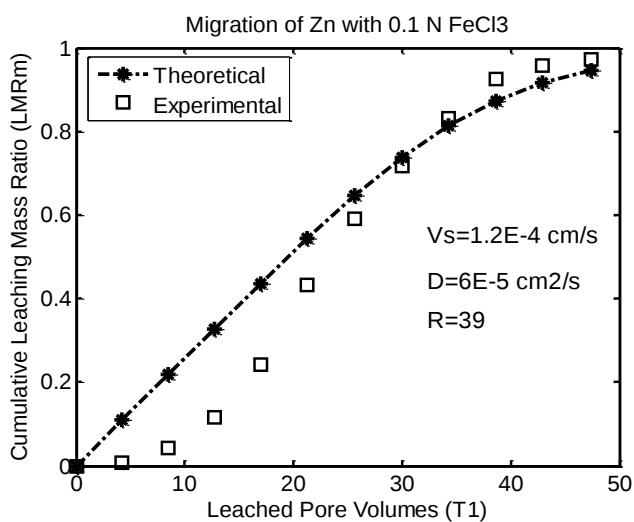


Fig 9 Theoretical elution curve of Zinc with 0.1 N FeCl₃

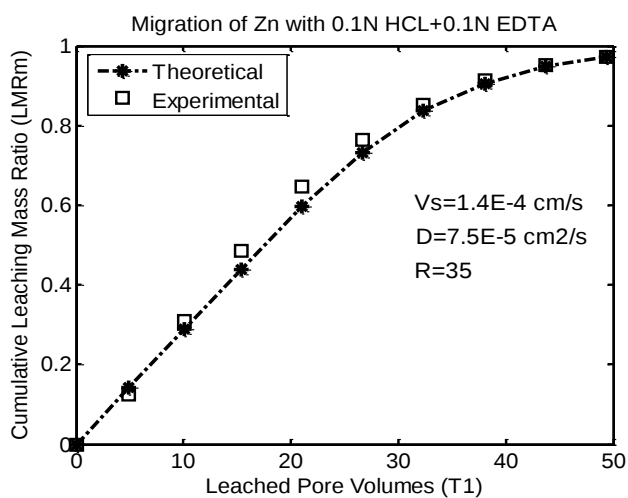


Fig 10 Theoretical elution curve of Zinc with 0.1 N FeCl₃

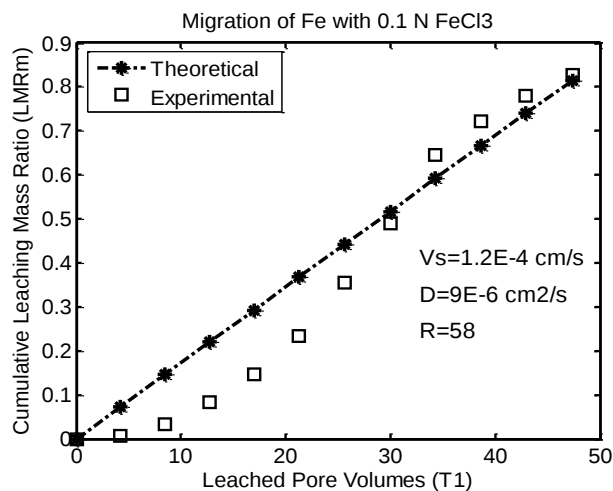


Fig 11 Theoretical elution curve of Iron with 0.1 N FeCl_3

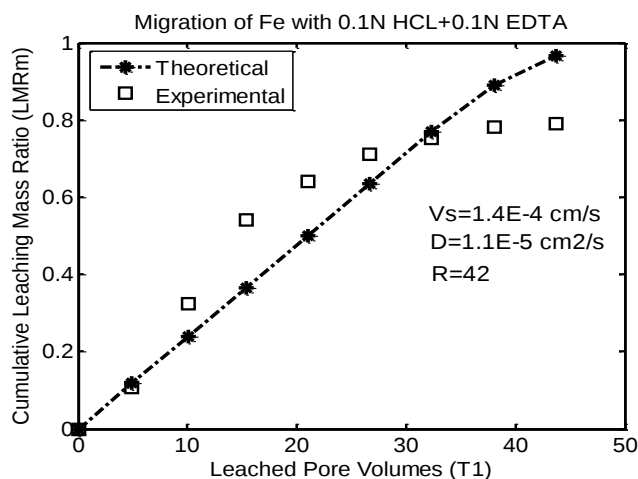


Fig 12 Theoretical elution curve of Iron with 0.1 N HCl + 0.1 N EDTA

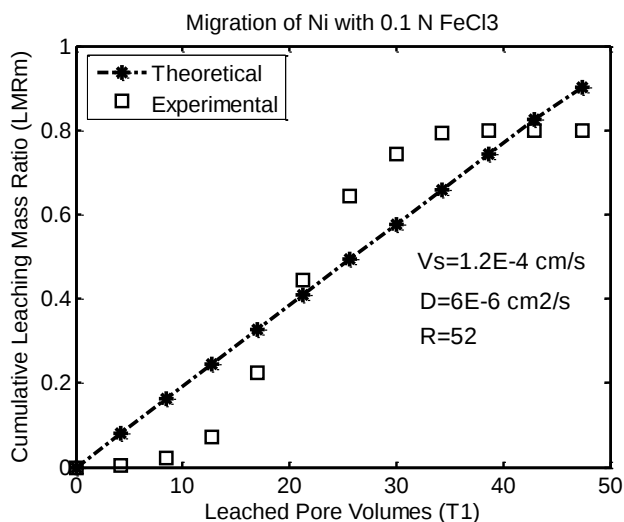


Fig 13 Theoretical elution curve of Nickel with 0.1 N FeCl_3

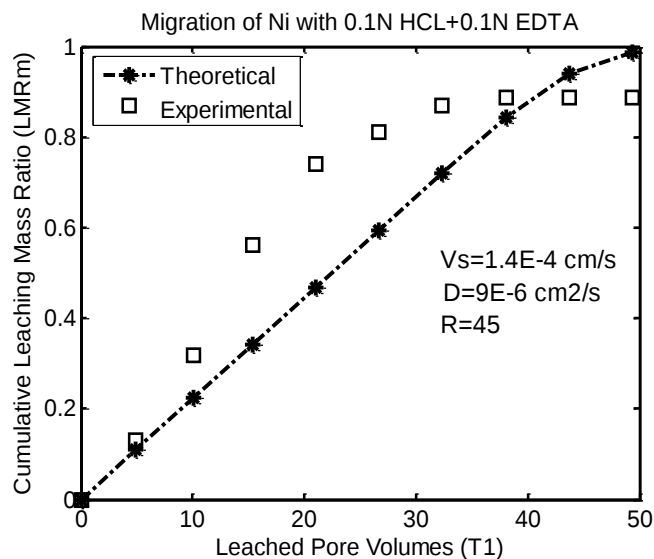


Fig 14 Theoretical elution curve of Nickel with 0.1 N HCl + 0.1 N EDTA

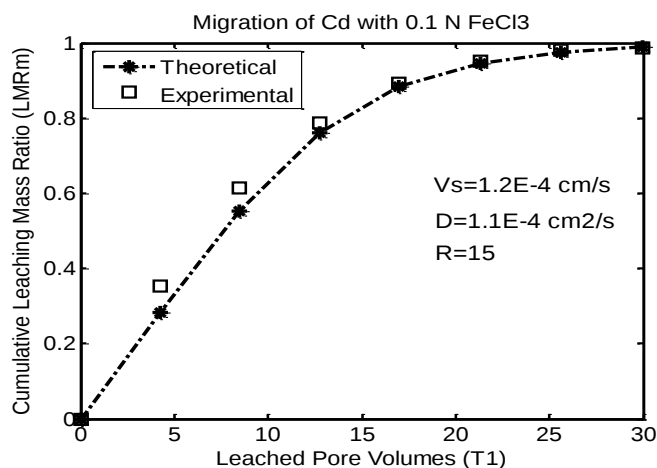


Fig 15 Theoretical elution curve of Cadmium with 0.1 N FeCl₃

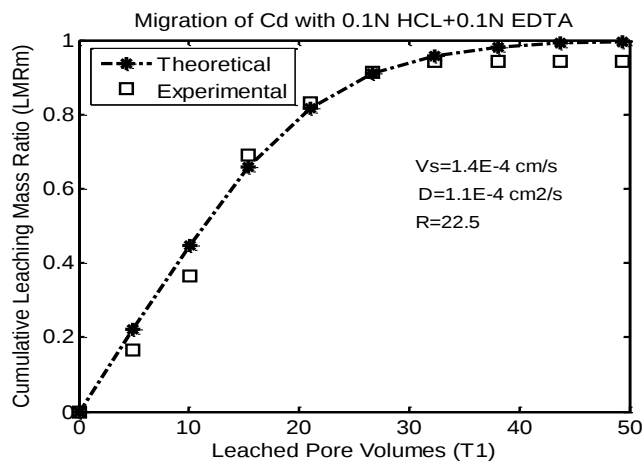


Fig 16 Theoretical elution curve of Cadmium with 0.1 N HCl + 0.1 N EDTA

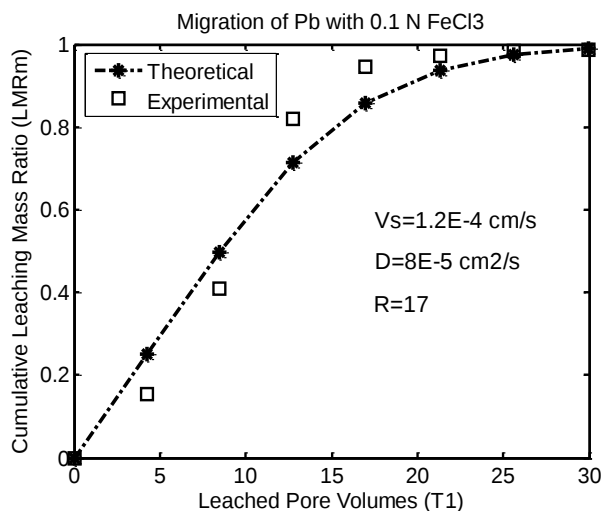


Fig 17 Theoretical elution curve of Lead with 0.1 N FeCl₃

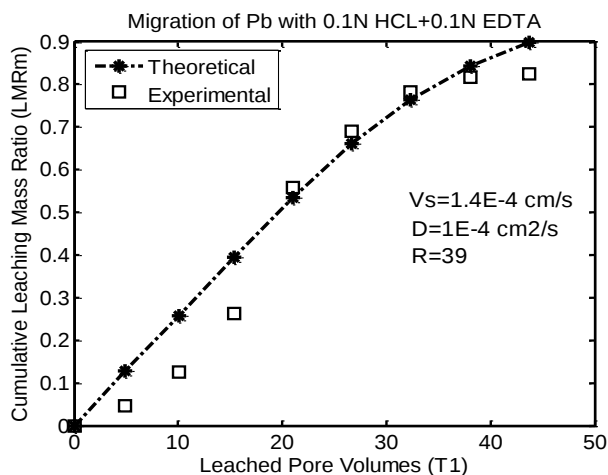


Fig 18 Theoretical elution curve of Lead with 0.1 N HCL + 0.1 N EDTA

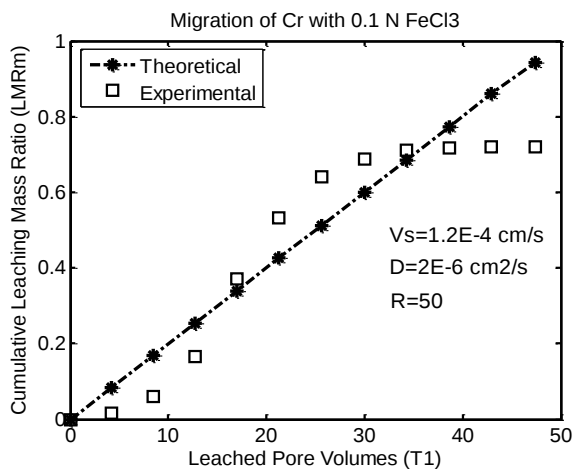


Fig 19 Theoretical elution curve of Chromium with 0.1 N FeCl₃

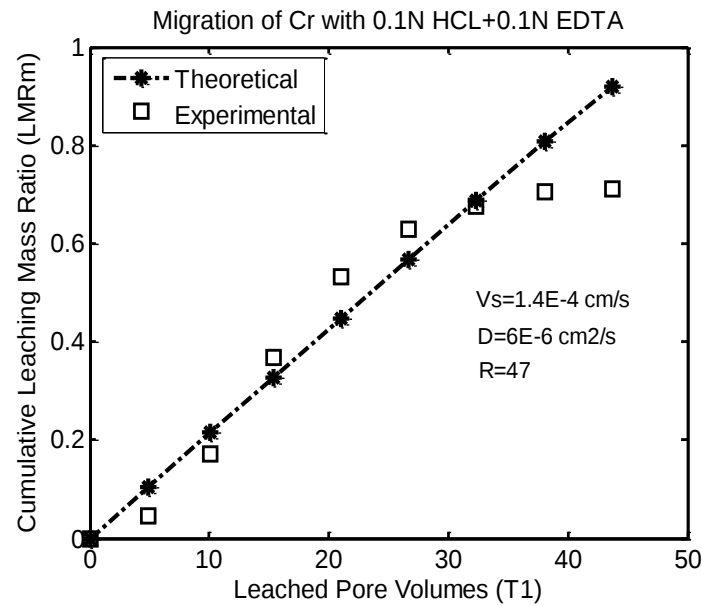


Fig 20 Theoretical elution curve of Chromium with 0.1 N HCl + 0.1 N EDTA

Table 3. Contaminant transport parameters of different metal ions

Pollutant	With FeCl ₃		With 0.1 N HCl + 0.1 N EDTA	
	Dispersion coefficient (D) (cm ² /s)	Retardation Factor (R)	Dispersion coefficient (D) (cm ² /s)	Retardation Factor (R)
Copper (Cu)	1.1 x 10 ⁻⁵	41.5	1.5 x 10 ⁻⁵	39.0
Zinc (Zn)	6.0 x 10 ⁻⁵	39.0	7.5 x 10 ⁻⁵	35.0
Iron (Fe)	9.0 x 10 ⁻⁶	58.0	1.1 x 10 ⁻⁵	42.0
Chromium (Cr)	2.0 x 10 ⁻⁶	50.0	6.0 x 10 ⁻⁶	47.0
Cadmium (Cd)	1.1 x 10 ⁻⁴	15.0	1.1 x 10 ⁻⁴	22.5
Nickel (Ni)	6.0 x 10 ⁻⁶	52.0	9.0 x 10 ⁻⁶	45.0
Lead (Pb)	8.0 x 10 ⁻⁵	17.0	1.0 x 10 ⁻⁴	39.0

ANALYSIS OF RESULTS

The % removal of metal ions with different leaching solutions have been compared and presented in Figs 21 – 27. It can be observed that Cu, Cd, Pb, Fe and Cr have got higher removal efficiencies with FeCl₃ as leaching solution whereas Zn and Ni got their higher removal efficiencies with HCl + EDTA. The removal efficiencies of FeCl₃ and HCl + EDTA

were almost same for most of the metals. Almost all the metals have attained more than 90% removal efficiency except Fe and Cr. The highest removal efficiency of Fe and Cr were 82.8% and 72.1% respectively with FeCl₃ as leaching solution.

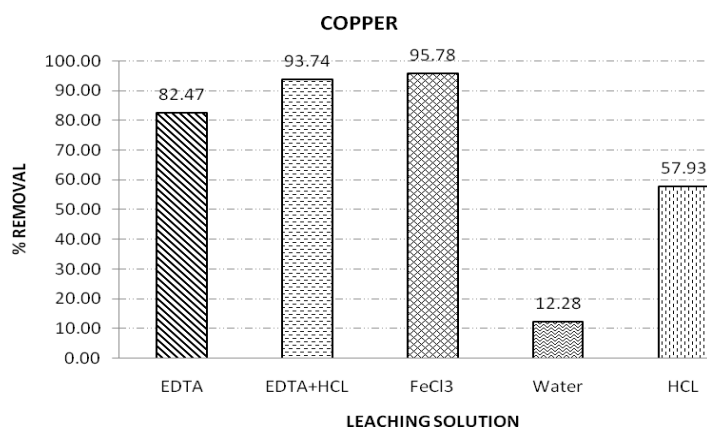


Fig 21 Removal efficiencies of different leaching solutions for Copper

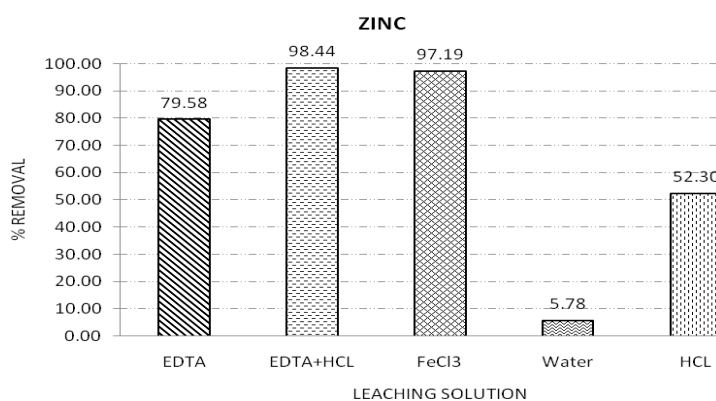


Fig 22 Removal efficiencies of different leaching solutions for Zinc

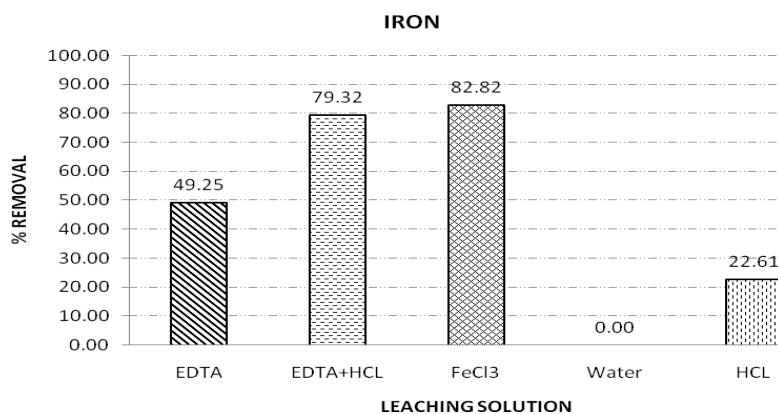


Fig 23 Removal efficiencies of different leaching solutions for Iron

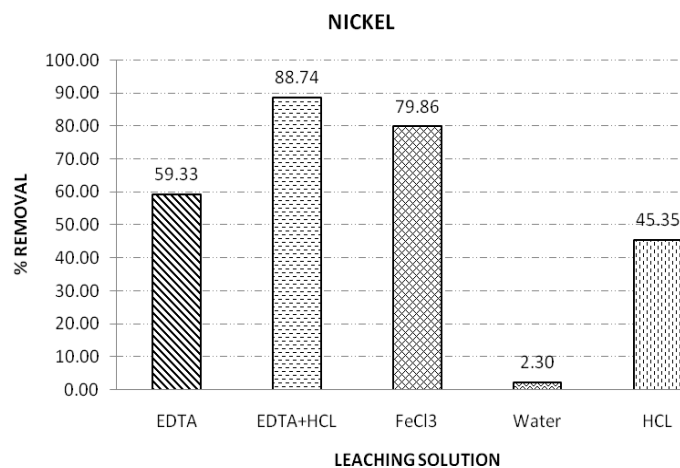


Fig 24 Removal efficiencies of different leaching solutions for Nickel

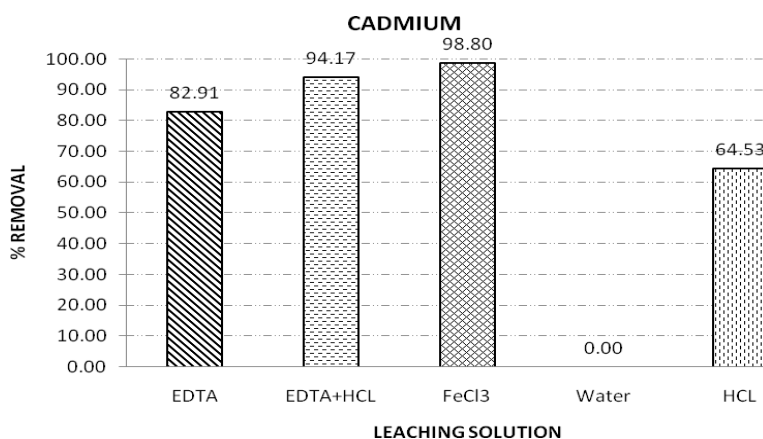


Fig 25 Removal efficiencies of different leaching solutions for Cadmium

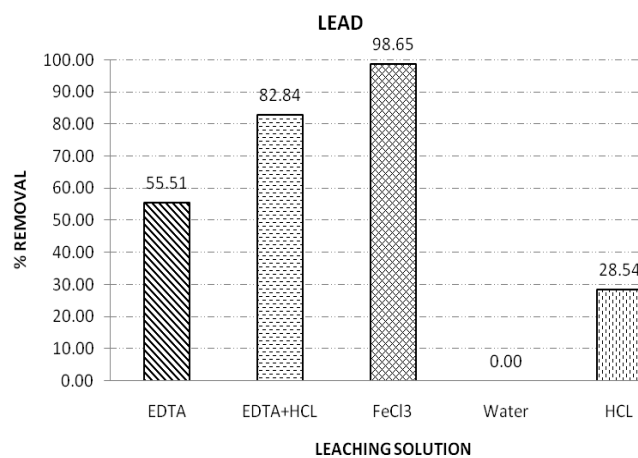


Fig 26 Removal efficiencies of different leaching solutions for Lead

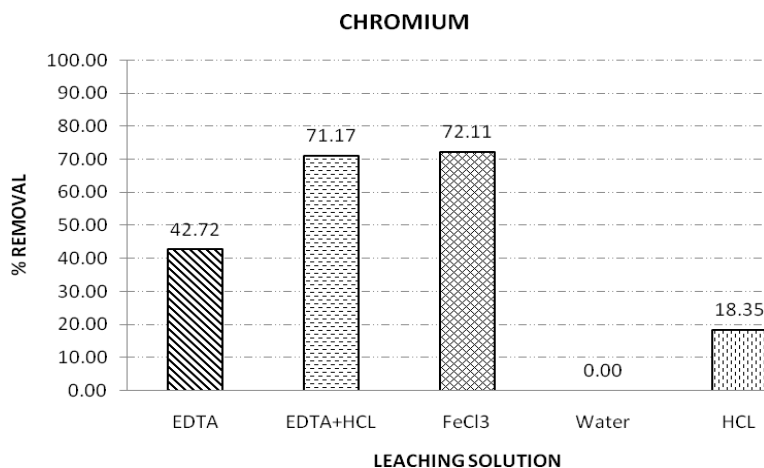


Fig 27 Removal efficiencies of different leaching solutions for Chromium

CONCLUSIONS

- 1 The removal efficiencies of distilled water, 0.1 N HCl, 0.1 N EDTA, 0.1 N HCl + 0.1 N EDTA and 0.1 N FeCl₃ were determined to remove the metal ions Cu, Zn, Fe, Ni, Cd, Pb and Cr present in the ETP Sludge.
- 2 It was found that the removal efficiencies of 0.1 N FeCl₃ were higher than the other solutions used in this study. But the removal efficiencies of 0.1 N FeCl₃ and 0.1 N HCl + 0.1 N EDTA were almost same and hence they can be suggested as the best leaching solutions for this sludge.
- 3 The % removals obtained with different leaching solutions are as follows.

Distilled water: Cu (12.3%) > Zn (5.8%) > Ni (2.3%) > Cd (0%) > Pb (0%) > Fe (0%) > Cr (0%)

0.1 N HCL: Cd (64.5%) > Cu (57.9%) > Zn (52.3%) > Ni (45.3%) > Pb (28.5%) > Fe (22.6%) > Cr (18.3%)

0.1 N EDTA: Cd (82.9%) > Cu (82.5%) > Zn (79.6%) > Ni (59.3%) > Pb (55.5%) > Fe (49.3%) > Cr (42.7%)

0.1 N HCL + 0.1 N EDTA: Zn (98.4%) > Cd (94.2%) > Cu (93.7%) > Pb (82.8%) > Ni (88.7%) > Fe (79.3%) > Cr (71.2%)

0.1 N FeCl₃: Cd (98.8%) > Pb (98.7%) > Zn (97.2%) > Cu

- (95.8%)>Fe (82.8%)>Ni
(79.9%)>Cr (72.1%)
- 4 The transport parameters of different metal ions were found by comparing the theoretical elution curves with experimental elution curves.
 - 5 The Dispersion coefficients of different metals were in the range of 2.0×10^{-6} - 1.1×10^{-4} cm²/s and Retardation factors were in the range of 15 – 58.
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