

# The Influence of Acids and pH in Removal Efficiency of Heavy Metals by Inorganic Coagulants

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## ABSTRACT

Chemical precipitation/coagulation using two inorganic coagulants (aluminium sulphate [ $\text{Al}_2(\text{SO}_4)_3$ ] and calcium oxide (CaO)) was studied to remove heavy metals (HMs) from soil leachate using three acids ( $\text{HNO}_3$ , HCL, and  $\text{H}_2\text{SO}_4$ ) to adjust the pH of the leachate to 2.0, 3.0, and 5.0, as appropriate. Results obtained with atomic absorption spectrophotometry (AAS) showed that  $\text{Al}_2(\text{SO}_4)_3$  coagulated Cd totaling 44, 72 and 95% at pH 2.0, 34, 53 and 48% at pH 3.0, and 79, 98 and 92% at pH 5.0 using the above three acids. CaO also coagulated Cd (76, 76, 86% at pH 2.0; 98, 97, 98% at pH 3.0; 78, 80, 84% at pH 5.0) making Cd the most coagulated metal. Cu (12% at pH 2.0, 5% at pH 3.0 and 8% at pH 5.0) was the least coagulated metal using HCL with CaO. Overall, the coagulation of metals was highest in all three acids at pH 5.0.

**Keywords:** aluminum sulphate, calcium oxide, effluent, flocculation, leachate, precipitate

## INTRODUCTION

The contribution of domestic and industrial input into the sewage system is one reason why the concentration of heavy metals (HMs) in soil and sewage sludge (SS) varies from place to place. HMs are tightly bound to organic solids and minerals making it very difficult to be separated by any physical separation process. HMs are solubilized from sludge solids by an acidification process (Ukiwe and Oguzie 2008). The resultant liquid containing the solubilized metals is then separated from the suspended solids fraction by a physical separation step. Chemical precipitation is further applied to convert the soluble metallic ions to insoluble metallic forms. The subsequent removal of the precipitate formed from the resultant liquid is through any physical separation process which can include: membrane filtration, sedimentation, flotation, etc. In HM precipitation, the addition of reagent must be carefully monitored to prevent excess build-up in sludge. Efficiency of the system relies on an adequate solid separation process which could be costly depending on the reagents used, the required system controls, and the involvement of an operator in system operation. Performance of a membrane bioreactor in removal of HMs from industrial wastewaters was studied by Moslehi *et al.* (2008). According to their study, chromium with a concentration below 50 mg/l had a removal efficiency of about 95% while the activated sludge process showed poor removal efficiency in the case of zinc (Zn), but when the activated sludge process was used in conjunction with the membrane process, the removal efficiency of Zn increased to 76%. The study noted that the membrane process showed an improvement of efficiency of about 44-65% in extracting lead (Pb) at 50 mg/l. Studies on historical changes of HM content and sequential extraction in a sediment core from the Gorgan Bay revealed that HM content increased towards the top of the sediment core, while chemical partitioning studies also revealed that percentiles and amounts of Zn, nickel and cobalt in the non-lithogenous phase of the sample increased slightly towards the top of the core sediment (Kabassi *et al.* 2008). Sorption from an aqueous solution onto limestone fines followed by flotation

was demonstrated to be a simple, rapid and economic procedure for removing Pb (II) from aqueous solutions. Ghazy *et al.* (2007) revealed that 99% of Pb (II) ions were removed from aqueous solutions at pH 7 after shaking for 5 min at 25°C. The study suggested that the sorption-flotation procedure could be applied successfully in recovering Pb (II) ions from natural water samples.

Chemical precipitation could be performed together with coagulation to facilitate the binding of suspended and colloidal materials for ease of solubilization. The HM precipitation/coagulation process involves the addition of a chemical reagent to adjust the pH of the sludge which is dependent on the specific metal-reagent combination. At the desired pH, metals show minimum solubility in water and hence precipitate out of sludge solution. Alkalis, sulphides, and sulphates are principal reagents for the precipitation/coagulation process. A study comparing HM removal from acidic and saline soil leachate (SSL) using either electrochemical coagulation or chemical precipitation (Meunier *et al.* 2006) showed that the electrocoagulation process was evaluated via an electrolytic cell using mild steel electrodes whereas chemical precipitation was also evaluated using sodium/calcium hydroxide (NaOH/CaOH). By comparison, the electrocoagulation process was more effective than chemical precipitation in removing metals from SSL by varying the pH of SSL between 7 and 8 with a relatively low contamination level. However, both chemical precipitation processes (NaOH/CaOH) needed to be operated at higher pH values (pH 10) to be more effective in reducing metals from SSL. Chemical precipitation using NaOH/CaOH appears to be a predominant process of metal mobilization from soils in alkaline conditions especially when the concentration of metals ions is high (Yassen *et al.* 2006). The net effect of liming using NaOH/CaOH on metal transformation in soils depends on the relative changes in the pH and the increase in the concentration of calcium and sodium ions added through liming, a process which decrease adsorption precipitation of metals in soils at high pH (Yassen *et al.* 2006). Chemical coagulation of combined sewer overflow (CSO) to determine HM removal and treatment optimization have shown that by jar-testing with two com-

mercial coagulants (ferric and polyaluminium chloride), CSO samples collected as a function of time during various wet-weather events from inlets of Boudonville retention basin, Nancy, France, indicated efficient turbidity removal yielding effective HM elimination with both coagulants, although lower optimum dosages and higher restabilization concentrations were obtained with the aluminium-based coagulants (Samrani *et al.* 2008). The jar test to determine the HM concentration and to examine the application of the coagulation-flocculation process for the treatment of raw leachate showed that in optimum conditions, the removal efficiency of HM with ferric chloride is economical and cost benefit than the use of aluminium sulphate (Zazouli and Yousefi 2008). SS contains not only nutrient elements but also toxic elements like HMs. Accumulation of HMs in soil is due to long-term application of sludge to agricultural land. To establish the guidelines of SS application to agricultural land in terms of HM content forms and behavior in soil, the mobility and availability of HMs were analyzed in sludge using calcium hydroxide as the coagulant aid. Results showed that the application of sludge resulted in a pronounced increase in Zinc (Zn) and Cadmium (Cd) content, and a slight increase in Nickel (Ni) and Copper (Cu) (Okamoto 2004). However, stabilization of HMs in ceramic (compound containing crocoites, chrome oxide, cadmium silicate, and copper oxide) made from wastewater treatment sludge and drinking water treatment sludge showed that the binding ability of HMs varies in coagulants such as silicate/aluminium oxides, acidic oxides, and basic oxides and that as the ratios of silicate and aluminium oxide decreases, the leaching contents of Cu and Pb increased, while the leaching contents of Cd and Cr first decrease and then increase. Nonetheless, under the variation of acidic oxides, leaching contents of Cd, Cu, and Pb increased, while Cr decreased (Xu *et al.* 2009). Biological municipal wastewater treatment can be adversely affected in a combined sewage system by highly loaded storm water with heavy rainfall (Park and Yoon 2003). To resolve this problem, a rapid coagulation process was introduced. The treatment capacity in the system was maximized by weighted coagulant additives sludge return and lamellar plates in a settling tank. Results of the jar test conducted found that the weighted coagulant additives primarily performed a role of particle settling for chemical flocculation in the coagulation step and sludge return was a predominant parameter to improve floc aggregation and its settling properties. Conclusively, the weighted coagulation process was proposed as one of the alternatives to economically treat HMs in heavily loaded rainfall events in municipal wastewater (Park and Yoon 2003). Solid waste leachates vary in their physical chemical and biological components. Studies of solid waste treatment have shown that anaerobic reactors with a detention time of 3 days had a 35% chemical oxygen demand (COD) removal in sequence batch reactors after chemical coagulation and clarification using ferric chloride. By increasing the detention time to 4.5 days, COD removal also increased by 45%. Meanwhile, nutrient adjustment with phosphorus and nitrogen increased the removal of effluent COD (Torabian *et al.* 2004).

The aim of the present study was to investigate the influence of acids and pH on the removal efficiency of HMs in soil leachate using inorganic coagulants such as aluminium sulphate  $[Al_2(SO_4)_3]$  and calcium oxide (CaO).

## MATERIALS AND METHODS

A sample of soil was collected from the dump site of the Federal Medical Center, Owerri, Nigeria, using a simple random sampling method. The soil sample was collected in a clean 5 kg polythene bag previously washed and rinsed with deionised water. The conventional 'Jar test' procedure was employed for coagulation and precipitation processes. Two coagulants were used in this research ( $Al_2(SO_4)_3$  and CaO).

For the  $Al_2(SO_4)_3$  coagulation test, about 50 g of soil sample was leached thoroughly with 500 ml deionised water in a 1000 ml

beaker for 30 min. The resultant leachate was centrifuged (MC Model 5415C) for 2 hrs at 200 rpm and allowed to stand for 1 hr. Three 100 ml beaker jars were set up at room temperature and separately for each jar; 20 ml each of the leachate was added followed by 2 g each of  $Al_2(SO_4)_3$  powder. The pH of the three solutions in the jars was separately adjusted to 2.0, 3.0, and 5.0 accordingly using NaOH/HCl as appropriate. Rapid churning of the mixtures was followed at 200 rpm for 5 min and then gentle mixing at 50 rpm for another 5 min. The flocs formed were allowed to settle for 1 hr and the supernatant was withdrawn from the beakers by using a three 10 ml plastic syringe each deep about 4 cm into the liquid in each jar. The withdrawn liquids were each digested separately using 10 ml each of  $HNO_3$  following the methods described by Ukiwe and Oguzie (2008). Each digestate was further analyzed using atomic absorption spectrophotometry (AAS Model SOLAAR V10) for the metals Cd, Mn, Pd, Ni, and Cu respectively, following the method described by Ukiwe and Oguzie (2008). Three repetitions were made for each beaker jar representing each pH level and the mean (mg/l) of HMs was obtained as described by Ukiwe *et al.* (2008). The above procedure was repeated as a control experiment without adding  $Al_2(SO_4)_3$ . The procedures as described above were also repeated using NaOH/ $HNO_3$  and NaOH/ $H_2SO_4$  to adjust the pH as appropriate as in above while using  $HNO_3$  for digestion. The entire procedures above were repeated using CaO as coagulant.

## Data analysis

Data were reported as arithmetic mean and standard deviation. BMDP statistical software was used for data analysis in this study. Analysis of variance (ANOVA) was employed to determine difference between mean concentration levels of HMs at various acids and pH levels. Moreover, the standard error of the difference between means and the generalized *t*-test were used to observe for significance in HM concentration in acids and pH levels at  $P < 0.01$ .

## RESULTS

The results obtained from this study are indicated in **Tables 1-6**. Metal precipitation from contaminated water involves the conversion of soluble HM salts to insoluble salts that will precipitate. The precipitate can then be removed from the treated water by physical methods such as clarification (settling) and/or filtration. The precipitation process usually uses pH adjustment, chemical precipitants, coagulants, and flocculants to increase particle size through aggregation. However, the process also generates very fine particles that are held in suspension by electrostatic surface charges. These charges cause clouds of counter-ions to form around the particles, giving rise to repulsive forces that prevent aggregation and reduce the efficiency of solid-liquid separation. Hence, chemical coagulants are often added to overcome the repulsive forces of the particles. Synthetic polyelectrolytes with anionic or cationic functional groups are some of the coagulants used to remove HMs under saline condition. An investigation by Hilal *et al.* (2008) proved that humic substances are easily coagulated using cationic metals and polyelectrolytes. The research showed that humic substance removal increased with increasing salinity and HM concentration and precipitation, while the addition of HMs in polyelectrolyte coagulation increased humic substance removal due to the combined charge neutralization of the metals and polyelectrolytes.

**Tables 1-6** show mean concentration (mg/l) of Cd, Mn, Pd, Ni, and Cu for  $HNO_3$ , HCL,  $H_2SO_4$  at pH 2.0, 3.0, and 5.0 respectively for soil leachate using  $Al_2(SO_4)_3$  and CaO coagulants.  $Al_2(SO_4)_3$  appears to be a better coagulant than CaO. Using  $Al_2(SO_4)_3$  as coagulant, Cd was coagulated (79, 98, 92%) using  $HNO_3$ , HCL, and  $H_2SO_4$  acids respectively at pH 5.0, though, CaO coagulated Cd better than  $Al_2(SO_4)_3$ ; (76, 76, 86%) at pH 2.0, (98, 97, 98%) at pH 3.0, and (78, 80, 84%) at pH 5.0, respectively. Nevertheless, it was noticed in this research that CaO poorly coagulated Cu in the range 5–18% using the above acids in the same order of pH 5.0 >

**Table 1** Concentration of heavy metals using aluminium sulphate as coagulant at pH 2.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control experiment
		Mean (mg/l) ± SD × 10 <sup>-1</sup>	Mean (mg/l) ± SD × 10 <sup>-1</sup>	Mean (mg/l) ± SD × 10 <sup>-1</sup>	Mean (mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0619 ± 0.1	0.0303 ± 0.2	0.0051 ± 0.0	0.1114 ± 0.0
Mn		1.8196 ± 0.0	0.0902 ± 6.3	0.1471 ± 5.2	5.6903 ± 0.0
Pd		0.3359 ± 0.2	0.4724 ± 1.6	0.1347 ± 0.7	2.4472 ± 0.0
Ni		0.2232 ± 1.9	0.1970 ± 2.0	0.2092 ± 2.9	0.2569 ± 1.2
Cu		0.0702 ± 2.4	0.0918 ± 1.0	4.2774 ± 0.1	5.1682 ± 0.0

**Table 2** Concentration of heavy metals using aluminium sulphate coagulant at pH 3.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control Experiment
		Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0304 ± 0.2	0.0216 ± 0.0	0.0238 ± 0.0	0.0466 ± 0.1
Mn		0.0259 ± 1.0	0.2237 ± 0.1	0.1210 ± 0.0	2.7820 ± 0.1
Pd		0.1450 ± 0.3	0.0036 ± 0.3	0.2920 ± 4.3	2.0206 ± 0.9
Ni		0.2600 ± 1.3	0.2473 ± 0.7	0.1346 ± 1.9	0.2794 ± 0.9
Cu		0.1086 ± 7.3	0.0407 ± 1.4	4.4370 ± 0.0	4.4742 ± 0.0

**Table 3** Concentration of heavy metals using aluminium sulphate coagulant at pH 5.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control Experiment
		Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0443 ± 0.1	0.0036 ± 0.0	0.0156 ± 0.0	0.2140 ± 0.2
Mn		0.6801 ± 1.0	0.1605 ± 0.1	0.6517 ± 0.0	4.3359 ± 0.1
Pd		0.0906 ± 0.0	0.1786 ± 3.5	0.1440 ± 2.8	1.4710 ± 0.6
Ni		0.2764 ± 2.4	0.2018 ± 4.0	0.1728 ± 1.3	0.3170 ± 1.0
Cu		0.0779 ± 1.2	0.0822 ± 0.4	4.7964 ± 0.2	4.8430 ± 0.1

**Table 4** Concentration of heavy metals using calcium oxide coagulant at pH 2.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control Experiment
		Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0242 ± 0.0	0.0245 ± 0.0	0.0145 ± 0.1	0.1050 ± 0.1
Mn		1.1995 ± 0.0	6.3570 ± 0.1	4.8350 ± 0.8	7.9605 ± 0.0
Pd		1.1833 ± 0.8	2.4606 ± 2.5	2.0295 ± 0.2	2.7598 ± 0.7
Ni		0.2216 ± 1.7	0.3802 ± 4.5	0.0152 ± 1.4	0.3978 ± 0.6
Cu		3.0911 ± 0.1	4.5879 ± 0.0	3.8401 ± 0.0	4.6450 ± 0.0

**Table 5** Concentration of heavy metals using calcium oxide coagulant at pH 3.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control Experiment
		Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0191 ± 0.0	0.0252 ± 0.0	0.0120 ± 0.0	1.0660 ± 0.8
Mn		0.0519 ± 1.0	4.1887 ± 1.0	0.4734 ± 0.2	8.4375 ± 0.0
Pd		1.8936 ± 0.6	1.9507 ± 0.3	1.7396 ± 0.2	2.3990 ± 0.1
Ni		0.1624 ± 4.9	0.1949 ± 1.1	0.0060 ± 0.3	0.2060 ± 0.6
Cu		3.7672 ± 0.0	4.3823 ± 0.1	3.4296 ± 0.0	4.6467 ± 0.0

**Table 6** Concentration of heavy metals using calcium oxide coagulant at pH 5.0.

Heavy metals	Acids	HNO <sub>3</sub>	HCL	H <sub>2</sub> SO <sub>4</sub>	Control Experiment
		Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>	Mean(mg/l) ± SD × 10 <sup>-1</sup>
Cd		0.0266 ± 0.0	0.0248 ± 0.0	0.0193 ± 0.0	0.1259 ± 0.0
Mn		2.8106 ± 5.2	4.9382 ± 0.0	5.5329 ± 0.8	5.8439 ± 0.1
Pd		2.2658 ± 1.4	2.1201 ± 1.8	1.6873 ± 0.1	2.7460 ± 0.3
Ni		0.3061 ± 4.4	0.1271 ± 3.2	0.0044 ± 1.4	0.3824 ± 1.6
Cu		3.5813 ± 0.0	4.0619 ± 0.1	3.6487 ± 0.2	4.4203 ± 0.0

pH 3.0 > pH 2.0. Also, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> poorly coagulated Cu (17, 8, and 9%) at pH 2.0, 3.0, and 5.0 when H<sub>2</sub>SO<sub>4</sub> was used to adjust the pH of the leachate. Coagulation peaked at pH 5.0 especially for Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> coagulant among the three acids.

The standard error of mean concentration (mg/l) of HMs at pH 2.0 using HNO<sub>3</sub> was 0.31 for Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 0.54 for CaO, while that of the control experiment (without coagulant) was 1.16 for Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 1.45 for CaO. This pH level and acid was used to note for significance in HMs present in the study. The standard error of the difference between mean levels of HM between HNO<sub>3</sub> and control experiment is 1.20 for Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 1.54 for CaO. The generalized *t*-test between HNO<sub>3</sub> (Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and CaO coagulants) and control experiments (without both coagulants) is 1.80 and 1.39, respectively by testing these values separately at 8 degrees of freedom each, *P* < 0.01.

## DISCUSSION

Precipitation of HMs has long been the primary method of treating metal-laden industrial wastewaters. As a result of the success of metals precipitation in such applications, the technique is being considered for use in remediating ground water containing HMs, including their radioactive isotopes. Chemical precipitation has its limitations which include careful and controlled reagent addition to preclude unacceptable concentrations in treatment effluent and further treatment of effluent as discharge standards become more stringent. To overcome these limitations, enhanced removal processes using coagulation and flocculation of polymeric origin have been studied. Results of jar test conducted to compare the metals removal efficiencies of the chemical treatment options using ferric chloride, alum, and anionic polymer showed that ferric chloride and anionic polymer

used together were more effective than only alum for metals removal (Johnson *et al.* 2008). Polymeric silicate colloids prepared by the hydrolysis of tetraethoxysilane and  $\text{NH}_4\text{OH}$  and modified with trimethoxysilane have been used successfully to adsorb HMs such as Pb and Cr in effluent (Li *et al.* 2004). After adsorption, the colloids are separated by coagulation with  $\text{Al}_2(\text{SO}_4)_3$ . This process removes HMs from effluent by up to 99%. Attempts have been made to examine the effectiveness of polymer addition to the coagulation process (Tatsi *et al.* 2003; Aguiler *et al.* 2005) and effectiveness of coagulation process during treatment of beverage industrial wastewaters to remove some of its trace metals (Amuda *et al.* 2006). Results obtained showed that combination of ferric chloride and polymer at different ratios achieved better removal efficiency of the metals than using only ferric chloride. Many treatment technologies for wastewaters containing HMs have been developed in recent years but poor removal efficiencies and complex operational procedures are some of the disadvantages encountered. Polyethyleneimine sodium xanthogenate (PEX) is a macromolecular HM coagulant prepared by grafting a xanthogenate group to polyethyleneimine used to counter poor removal efficiency of HMs in wastewaters. PEX is an amphoteric polyelectrolyte and at low pH the amino groups of PEX are electrically positive hence removing turbidity due to electroneutralization coagulation, but at higher pH values, both amino groups and xanthogenic radical groups contribute to the removal of Cu as a result of chelation (Chang and An 2007).

However, compared to traditional chemical methods of precipitation/coagulation using CaO and  $\text{Al}_2(\text{SO}_4)_3$ , the use of enhanced removal processes using polymers shows obvious advantages such as removal of turbidity and copper ions, higher removal efficiency, lower suitable pH value, and higher floc settlement velocity.

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