

Progressive Acidification: An Aspect of Chemical Leaching of Sewage Sludge

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Received: November 14, 2011

Accepted: December 1, 2011

Published: February 1, 2012

doi:10.5539/ijc.v4n1p62

URL: <http://dx.doi.org/10.5539/ijc.v4n1p62>

Abstract

The effect of progressive acidification using inorganic and organic acids in chemical leaching of sewage sludge was studied. Results of analyses using Atomic Absorption Spectrophotometer (AAS) showed that Mn was the most metal solubilized totaling 4.760 and 4.220 mg/l using 30 % (v/v) HNO₃ and 50 % (v/v) acetic acids respectively for digestion, while Cd (0.038 and 0.027 mg/l) was the least metal extracted using 10 % (v/v) H₂SO₄ and 50 % (v/v) benzoic acid respectively for digestion. HCl was generally observed to be the best extraction medium for the metals; Cd, Mn, Pb, Ni, and Cu at all acid range 10-90 % (v/v) concentrations. However, comparing the leaching capability of both acids type, the inorganic acids were observed to be more effective than organic acids in leaching heavy metals from sewage sludge.

Keywords: Absorption, Chelates, Concentration, Digestion, Extraction, Precipitation, Solubilization, Wastewaters

1. Introduction

Sewage sludge (SS) is produced from the treatment of wastewaters and vary depending on the source as different wastewater sources has different constituents with varying compositions in the sludge (UNEP, 2004). The amount of sewage produced and the population size density determine the method of treatment and disposal method of the sludge. The physiochemical processes involved in SS treatment tends to concentrate heavy metals (HMs) which are toxic or poisonous constituents in the sludge even in small amounts and which can only be removed by sludge management techniques such as acid solubilization.

SS contains nutrients, organic matter, pathogens and contaminants ranging from suspended large solids through colloids to dissolved solids which have to be treated and processed to avoid prospective health risks when disposed off (Harrison & Oakes, 2002). Processing of SS includes treatment processes that involve settling of sludge to remove solids such as fats, oils etc. A study conducted on sewage processing noted that screening, grit removal and grease removal are the first steps of pre-treatment where the solids are removed from the effluent sewage water for disposal (UNEP, 2004). HMs are bound or incorporated into the organic matter, organic mineral aggregates and inorganic particles of the sludge and poses an obstacle to its utilization, making its

removal or extraction by physical means difficult (Morita & Tsuboi, 2000). The chemical form of the metals is a function of the method used to extract or reduce HMs from SS. These methods of extraction include bioleaching (using microbes) and chemical (using acids). Chemical extraction involves the use of both organic and inorganic acids to extract HMs from SS. According to Yoshizaki & Tomida (2000), the sufficient removal of HMs from sludge can only be achieved by using acids as the extracting solution, though, report suggest that using acids as extracting solution could impart adversely on the environment (Ukiwe & Nwoko, 2010a). Nevertheless, recent studies have suggested organic acids as promising extracting agents since they are biodegradable and they yield high metal efficiency even at mildly acidic pH (Babel & Dacera, 2006). A study conducted on the solubility of metals in SS showed that HMs extraction from SS depended on factors such as pH, redox potential of the sludge, and the concentration of the metals and ligands (Naoum et al., 2001). Hence, in order to determine the extraction efficiencies of HMs from SS, Zhang et al. (2008) noted that in relation to the species of the metals in the sludge, the extraction efficiency depended on the sludge content, dosage of the extractants, reaction time and temperature.

Acid solubilization of HMs is a process of proton exchange from the acid to the sludge (Browski et al., 2004). After solubilization, the removal of the solubilized metals is achieved by chemical precipitation followed by a physical separation step (Stylianou et al., 2007). There are three major approaches available to reduce the concentration of HMs in SS (Marchioretto et al., 2002). Firstly, there should be control of industrial sources and other point sources where wastewaters discharge into sewer systems. Secondly, control of diffuse sources using lead (Pb) free gasoline and, thirdly, by the extractive removal of HMs from SS. The latter process makes use of extreme acidic conditions for sufficient solubilization and release of metals to the liquid medium. The liquid containing the solubilized metals could be separated by centrifugation.

An investigation into the effect of aeration and acidification on HMs solubilization of SS using the various experimental procedures which include; aeration before acidification and aeration after acidification demonstrated that Mn and Cu were the highest metals extracted when the above experimental procedures were applied in metal extraction (Ukiwe & Nwoko, 2010b). However, when aeration was applied only, Cu extraction efficiency dropped remarkably. Ni and Pb were effectively extracted when the aeration after acidification protocol was used. The study further revealed that the extraction yield of Cd was least when the aeration before acidification procedure was employed. Some studies have suggested the application of aeration and acidification procedures to achieve best condition for HMs solubilization and extraction in sludge. These authors argue that mobility and bioavailability of metals depend on their chemical forms, as such, it is important to determine metal speciation while evaluating the amounts of metals leached under changing environmental conditions (Loska & Wiechula, 2006). However, aeration has been reported to cause an increase in the amount of Cu released from organically bound forms (labile organic material) and a decrease in the organic-sulphide fraction (stable organic material) in marine sediment slurries (Gerring, 1991).

When acid is added to sludge, the HMs present in the sludge dissolves and exists in solution (Arain et al., 2009). Optimum conditions for HMs removal from sludge can be achieved through variation of contact time between the extraction agent (acid) and the sludge solution. The effect of nitric acid concentration and sonication time on the removal efficiency of HMs from sludge was also investigated by Deng et al. (2009). Results obtained indicated that when nitric acid concentrations were varied from 0 to 0.65 M and sonication time also varied from 0 to 29 min, the removal efficiency of Cu, Zn, and Pb increased with increasing nitric acid concentration and sonication time. The study also noted that sonication time of 20 min resulted in maximum HM removal efficiency. Ukiwe and Iwu (2010) attempted an investigation into the effect of reaction time and acids in chemical leaching of HMs in SS and their study revealed that effective and efficient metal extraction could be achieved when acid and sludge are reacted for a contact time of 1 hr. Though a contact time of 6 hrs led to poor metal extraction. Also, there was no observable trend in HM extraction as contact time was ascended from 1, 2, 4 and 6 hrs respectively. Various complexing agents such as EDTA (ethylenediamine tetracetic acid), EDDS (ethylenediamine disuccinic trisodium salt), DTPA (diethylenetriamine pentacetic acid), and NTA (nitrilotriacetic acid) have been applied to enhance metal solubility and extraction in sludge system (Ukiwe et al., 2010). Several factors should be considered when comparing chelating agents for HM decontamination. These factors include among others; the ratio of chelating/HMs (Poletini et al., 2008). Using smaller quantities of chelating agents generates competition of chelates with other complex substances found in the sludge. Addition of EDTA to sludge base on stoichiometric requirements for metals such that the percentage of metal removal by EDTA do not correspond to stability constants for the EDTA complex raises issues associated with HMs bound by various insoluble salts. EDDS has been shown to have greater rate of degradation and strong chelating characteristics than other chelating agents. Nevertheless, its biodegradation rate varies largely depending upon

the examined conditions (Evangelou et al., 2007). Interaction experiments have demonstrated that EDTA and NTA are more efficient than malate and citrate in solubilizing metals with minimal difference in extraction efficiency between the two chelates (Peñolosa et al., 2007). Consequently, when complexing agents such as citric and oxalic acids as well as EDTA are applied to sludge, metals such as Cr could be solubilized at a higher pH (3-5) than when a strong acid such as HNO₃ is applied.

The aim of the present study was to evaluate the effect of progressive acidification using inorganic and organic acids in chemical leaching of domestic sewage sludge.

2. Materials and Method

The sewage sample used in this study was obtained from the male hostel septic tank of the Federal University of Technology, Owerri, Nigeria. The septic tank has a capacity of about 800 m³/day of sludge. Two 100 L plastic containers previously washed and rinsed with deionised water were used to collect the sewage sample from the tank. The containers were left to stand for 24 hrs in the Project Laboratory of the Department of Chemistry, Federal University of Technology, Owerri, Nigeria. These samples form the stock solution. From these stock solutions, samples were continuously drawn for the experiment. A quantity of sample equaling 2 L of sewage was measured into a 2 L beaker previously washed and rinsed with deionised water. About 1 L of this sewage sample was measured into another 2 L conical flask and centrifuged (MC 5415C) for 2hrs at 150 rpm. The resulting solution was filtered through Whatman No. 42 filter papers. To 30 ml of the filtrate in a 100 ml beaker, 10 ml of a 10 % (v/v) HNO₃ solution was added and the mixture stirred continuously for 5 min, while the pH of the mixture was maintained at 3.0 with appropriate addition of 10 M NaOH solution. The mixture was then heated at 80 °C for 1 hr with intermittent addition of deionised water to prevent the mixture from drying up. The mixture was cooled and filtered again through Whatman No. 42 filter papers. The resulting solution was made up to 100 ml with deionised water in a 100 ml standard flask and 20 ml of this solution was used for analyses by Atomic Absorption Spectrophotometer (AAS SOLAAR V10) for the elements Cd, Mn, Pb, Ni, and Cu respectively. Three repetitions were made and the mean concentration (mg/l) of HMs was obtained by the method described by Ukiwe & Oguzie (2008). The above procedure was repeated using 30, 50, 70, and 90 % (v/v) HNO₃ respectively. However, the entire procedure was also repeated using 10, 30, 50, 70, and 90 % (v/v) H₂SO₄, HCl, citric, acetic and benzoic acids respectively.

3. Statistical Analysis

Data are given as arithmetic mean and standard deviation. The *t*-test was used to estimate significant difference in mean concentration using HCl as study digestion medium.

4. Results and Discussion

Table 1-6 give mean concentration (mg/l) of Cd, Mn, Pb, Ni, and Cu for acid digestion of SS using inorganic acids (HNO₃, H₂SO₄, and HCl), organic acids (citric, acetic and benzoic) at various concentrations. With the inorganic acids, Mn (4.760 mg/l) was the most mobilized metal using 30 % (v/v) HNO₃. Mn also appear to be the overall highest metal extracted totaling (2.390, 4.760, 4.240, 4.450 and 2.120 mg/l) using 10, 30, 50, 70, and 90 % (v/v) HNO₃ respectively. Cd was the least metal extracted using the inorganic acids. Cd extraction using 10 % (v/v) HNO₃, H₂SO₄ and HCl was 0.050, 0.038, and 0.039 mg/l respectively. HCl was generally observed to be the best medium for extraction of Pb, Ni, and Cu using both acids type at all acid concentrations, though the extraction of HMs wasn't significant between acid ranges. This is because the *t*-test between 10 and 30 %, 30 and 50 %, 50 and 70 % as well as 70 and 90 % (v/v) acid concentrations are 0.14, 2.71, 3.59, and 2.27 respectively. Test these values separately at 8 degrees of freedom, *P* < 0.01.

With the organic acids, Mn was the overall highest metal extracted. The extraction range with organic acids was 2.459, 2.420, 4.220, 1.987, and 2.425 mg/l respectively at 10, 30, 50, 70, and 90 % (v/v) acetic acid concentrations. Nevertheless, Cd was also the least metal extracted and extracted overall (0.027 mg/l) using benzoic acid at 50 % (v/v) acid concentration. It was noted in the present study that there wasn't any trend in HMs solubilization using the six acids types. HCl and acetic acids were observed to be the two most effective acids for metal extraction.

The extractive yield of HMs in sludge depend on the kind and concentration of acids used (Ukiwe & Oguzie, 2008). Metals can be removed from sludge by acid leaching (chemical treatment). In SS metals occur in inorganic or organic complexes. In order to assess their mobility and the hazards they pose, it is necessary to distinguish their forms and assay their quantities especially the extreme toxic ones such as Pb, Cd, and Zn. The most popular chemical extraction procedures involve treating a sample of the sludge with chemical solutions of various leaching strength (Turek et al., 2005). It has been reported that the release of HMs from SS during

treatment with chemical reagent is strongly dependent on pH (Rajczyk & Wiśniowska, 2005). Extraction of metals from low-grade ores, mining wastes, and SS using organic acid excreted by heterotrophic organisms was studied by Jain & Sharma (2004). The study noted that there exist a similarity between electronic wastes and other metal containing wastes. Hence, it is expected that organic acids could also be used to leach metals from electronic wastes. Leaching capability of citric, malic, gluconic and lactic acids as well as bromine in dissolving metallic fractions of printed circuit board (PCB) wastes has been studied by Chien et al. (2000). It was revealed that the intrinsic potency of organic acids in leaching metallic fraction require decoupling the effects of secondary reactions from metal dissolution. Adsorption was shown to be the most common reaction hampering metal dissolution. However, several factors were noted to influence adsorption reaction. These include surface charge of the non-metallic fractions in PCB, metal speciation, solution pH, and the nature of ligands in solution. The study further revealed that although adsorption could not be completely overcome, its effect could be sufficiently suppressed by altering the solution pH during leaching thereby enhancing metal recovery. Removal of HMs from sludge before disposal or application to farmland is a necessary step to achieve a more safe sludge usage or disposal. Tu et al. (2011) also investigated a novel approach for leaching HMs from sludge fractions. It was shown that radiation time, power and energy had immense effect on microwave assisted extraction of HMs from SS using HEDTA (hydroxyethylenediaminetriacetic acid) and oxalic acid. By applying the Tessier's Sequential Extraction (TSC) procedure, concentration of extractable fractions of Cu, Ni, and Cr in sludge were increased when the microwave time, power intensity and energy density were increased. The application of chemical extraction as a part of pre-treatment method is a feasible option. A recent study examining chemical leaching process in assessing the mobility of Zn, Cu, Cr, Ni and Pb using inorganic acids (HNO₃, HCl) and organic acids (citric, oxalic) reported that solubilization of metals using inorganic acids achieved maximum extraction efficiency at pH value lower than 2.0 and acid contact times of 1hr. However, using oxalic acid didn't show high extracting efficiency compared to citric acid at pH 2.0 and 1hr contact time. It was further noted that citric acid appeared to be highly effective in extracting Cu and Zn at longer leaching contact times. But at pH 3.0, the extracting efficiency of Cr and Cu using citric acid was relatively high (Gaber et al., 2011). These results agree with the results of the present study which noted that inorganic acids were more effective in leaching HMs from SS.

5. Conclusion

Extraction of HMs from SS before disposal or application to farmland is a necessary step for proper sludge management. Inorganic acids have been tried successfully due to their high leaching capability, though; these acids have associated problems with environmental contamination. Nevertheless, organic acids are becoming attractive agents to overcome the problem encountered using inorganic acids for metal leaching for the following reason; organic acids are mildly acidic and biodegradable and condition pertinent for high efficiency metal solubilization. The present study has demonstrated the need for further research using organic acids to leach HMs from SS.

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Table 1. Concentration (mg/l) of HMs in sewage digestate using HNO₃

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50 % (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.050 ± 0.0	0.050 ± 0.0	0.060 ± 0.0	0.050 ± 0.0	0.040 ± 0.0
Mn	2.390 ± 0.2	4.760 ± 0.3	4.240 ± 0.1	4.450 ± 0.8	2.120 ± 0.5
Pb	0.110 ± 0.0	0.090 ± 0.0	0.120 ± 0.0	0.082 ± 0.0	0.040 ± 0.0
Ni	0.080 ± 0.0	0.060 ± 0.0	0.090 ± 0.0	0.090 ± 0.0	0.140 ± 0.0
Cu	0.140 ± 0.0	0.010 ± 0.0	0.280 ± 0.0	0.130 ± 0.0	0.100 ± 0.0

Table 2. Concentration (mg/l) of HMs in sewage digestate using H₂SO₄

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50 % (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.038 ± 0.0	0.044 ± 0.0	0.051 ± 0.0	0.056 ± 0.0	0.045 ± 0.0
Mn	1.619 ± 0.5	2.683 ± 0.6	2.981 ± 0.6	2.087 ± 0.5	3.013 ± 0.7
Pb	0.766 ± 0.1	0.307 ± 0.0	0.042 ± 0.0	0.421 ± 0.1	1.139 ± 0.3
Ni	0.048 ± 0.0	0.084 ± 0.0	0.054 ± 0.0	0.049 ± 0.0	0.255 ± 0.0
Cu	0.219 ± 0.0	0.476 ± 0.1	0.433 ± 0.1	0.337 ± 0.1	0.453 ± 0.1

Table 3. Concentration (mg/l) of HMs in sewage digestate using HCl

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50 % (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.039 ± 0.0	0.042 ± 0.0	0.041 ± 0.0	0.042 ± 0.0	0.046 ± 0.0
Mn	2.834 ± 0.7	2.587 ± 0.7	3.491 ± 0.5	2.776 ± 0.5	3.582 ± 0.6
Pb	3.516 ± 0.6	3.722 ± 0.8	3.661 ± 0.2	3.462 ± 0.5	3.557 ± 0.2
Ni	2.456 ± 0.4	2.134 ± 0.3	2.684 ± 0.2	2.087 ± 0.8	2.662 ± 0.2
Cu	1.983 ± 0.6	2.272 ± 0.7	2.895 ± 0.7	1.222 ± 0.6	1.769 ± 0.2

Table 4. Concentration (mg/l) of HMs in sewage digestate using acetic acid

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50 % (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.034 ± 0.0	0.044 ± 0.0	0.028 ± 0.0	0.043 ± 0.0	0.036 ± 0.0
Mn	2.459 ± 0.1	2.420 ± 0.4	4.220 ± 0.7	1.987 ± 0.6	2.425 ± 0.5
Pb	0.273 ± 0.1	0.237 ± 0.2	0.241 ± 0.0	0.203 ± 0.0	1.185 ± 0.7
Ni	0.104 ± 0.0	0.082 ± 0.0	0.086 ± 0.0	0.082 ± 0.0	0.190 ± 0.0
Cu	0.519 ± 0.1	0.788 ± 0.1	0.223 ± 0.0	0.630 ± 0.1	0.558 ± 0.1

Table 5. Concentration (mg/l) of HMs in sewage digestate using benzoic acid

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50 % (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.047 ± 0.0	0.055 ± 0.0	0.027 ± 0.0	0.036 ± 0.0	0.041 ± 0.0
Mn	0.180 ± 0.0	0.203 ± 0.0	0.225 ± 0.0	0.385 ± 0.1	0.350 ± 0.6
Pb	0.345 ± 0.0	0.377 ± 0.2	0.398 ± 0.3	0.387 ± 0.1	0.451 ± 0.5
Ni	0.276 ± 0.0	0.246 ± 0.1	0.239 ± 0.1	0.219 ± 0.0	0.214 ± 0.0
Cu	0.189 ± 0.0	0.269 ± 0.2	0.240 ± 0.0	0.235 ± 0.0	0.248 ± 0.1

Table 6. Concentration (mg/l) of HMs in sewage digestate using citric acid

Extractant concentration Heavy metals	10 % (v/v)	30 % (v/v)	50% (v/v)	70 % (v/v)	90 % (v/v)
	Mean (mg/l) ± SD				
Cd	0.050 ± 0.0	0.070 ± 0.0	0.050 ± 0.0	0.050 ± 0.0	0.060 ± 0.0
Mn	0.103 ± 0.0	0.137 ± 0.0	0.174 ± 0.1	0.379 ± 0.3	0.293 ± 0.1
Pb	0.423 ± 0.7	0.216 ± 0.0	0.285 ± 0.3	0.479 ± 0.4	0.488 ± 0.7
Ni	0.100 ± 0.0	0.090 ± 0.0	0.050 ± 0.0	0.100 ± 0.0	0.090 ± 0.0
Cu	0.143 ± 0.0	0.168 ± 0.0	0.127 ± 0.0	0.213 ± 0.0	0.249 ± 0.2