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Autoheated thermophilic aerobic sludge digestion and metal bioleaching in a two-stage reactor system

Rohan Jain[1], Ashish Pathak[2], T. R. Sreekrishnan[1], M. G. Dastidar[2,*]

1. Department of Biochemical Engineering & Biotechnology Indian Institute of Technology, Hauz Khas, New Delhi-110016, India.
E-mail: rohanjain.iitd@gmail.com
2. Centre for Energy Studies, Indian Institute of Technology, Hauz Khas, New Delhi-110016, India

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Abstract

A two-stage process has been developed for stabilization of sludge and removal of heavy metals from the secondary activated sludge with high rate of energy and time conservation. The first stage of the process involves autoheated thermophilic aerobic digestion at 55–60°C inoculated with less-acidophilic thermophilic sulfur-oxidizing microorganisms (ATAD). The results show that it is possible to maintain the autoheated conditions (55–60°C) in the ATAD reactor up to 24 hr, leading to reduction of 21% total solids (TS), 27% volatile solids (VS), 27% suspended solids (SS) and 33% volatile suspended solids (VSS) from the sludge. The sludge pH also decreased from 7 to 4.6 due to the activity of less-acidophilic thermophilic microorganisms. In the second stage operation, the digested sludge (pH 4.6, TS 31.6 g/L) from stage one was subjected to bioleaching in a continuous stirred tank reactor, operated at mean hydraulic retention times (HRTs) of 12, 24 and 36 hr at 30°C. An HRT of 24 hr was found to be sufficient for removal of 70% Cu, 70% Mn, 75% Ni, and 80% Zn from the sludge. In all, 39% VSS, 76% Cu, 78.2% Mn, 79.5% Ni and 84.2% Zn were removed from the sludge in both the stages.

Key words: bioleaching; thermophilic aerobic digestion; heavy metals; sludge

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Introduction

Treatment of municipal wastewater results in the generation of a huge amount of sludge, the disposal of which is a serious environmental problem. For disposal of such large quantities of sludge, the most commonly used practice around the world is land application. This is being considered as one of the most economical methods of sludge disposal because the sludge is freely and easily available nutrient source and hence can save substantial cost, if applied to soil as a fertilizer (Metcalf and Eddy, 2003). However, the presence of putrescible organic matter, pathogens and heavy metals in the sludge restricts its use as a fertilizer (Spinosa, 2007). Therefore, a sustainable sludge management programme will require stabilization of sludge and removal of heavy metals prior to its disposal on land.

Aerobic digestion at higher temperature (45–70°C) is considered to be an attractive method for high rate removal of sludge solids and for reduction of pathogenic organisms in the sludge. The stabilized sludge is a well processed end product which is safe for land disposal (Kabrik and Jewell, 1982). However, to operate thermophilic digesters at 55–65°C, heat is to be supplied externally, which increases the process cost and limits its application for large scale operation. An alternative approach to solve this energy demanding problem is the autoheated thermophilic aerobic digestion (ATAD), in which the energy needed for maintaining the thermophilic conditions is produced by the aerobic digestion process itself. The auto-thermal conditions are achieved through the heat generated by the bio-oxidation of organic solids (Nosrati et al., 2004). This is possible by maintaining the minimum volatile suspended solids content of 2% in the feed sludge (Sreekrishnan et al., 2007). Autoheated thermophilic aerobic digestion produces biologically stabilized sludge while reducing both sludge mass and volume (Layden, 2007). The increased rate of degradation, pathogen inactivation, low hydraulic retention time and less demand of energy enhance the feasibility of ATAD for large scale application (Nosrati et al., 2007).

Besides solids and pathogen reduction, sludge also needs to be free of toxic metals. Bioleaching process using sulfur-oxidizing microorganisms has been reported to be an efficient and economical method for removal of heavy metals from the sludge (Pathak et al., 2008). The process has also been used successfully for various industrial sludges (Solisio et al., 2002; Zhou et al., 2006). The sulfur based bioleaching process using elemental sulfur as a cheap energy source produces acid which results in

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* Corresponding author. E-mail: mgdastidar@gmail.com

high solubilization of metals. Furthermore, bioleaching can be performed at neutral pH of the sludge under mesophilic conditions or mild thermophilic conditions, which provides an attractive opportunity to carry out sludge stabilization and metal bioleaching simultaneously (Shooner and Tyagi, 1995; Benmoussa et al., 1997). However, under mesophilic conditions sludge stabilization requires a longer time to obtain sufficient solid reduction. Under thermophilic conditions (55–60°C), although sludge stabilization and pathogen control take place very efficiently, bioleaching of heavy metals from sewage sludge is not an efficient process. The growth of the acidophilic microorganisms (*At. thiooxidans*) responsible for bringing down the pH (< 3) is not favored at high temperature (55–60°C), at which only the less-acidophilic microorganisms capable of bringing down the pH from 7 to 4.5 are able to grow.

In the present study, an attempt has been made to develop a two stage process for stabilization of sewage sludge and leaching of heavy metals from the sludge. In the first stage, the studies were conducted on ATAD supplemented with less-acidophilic thermophilic sulfur-oxidizing bacteria. At this stage, the sludge stabilization and lowering of pH from 7 to 4.5–5 were expected to take place due to the activity of autotrophic thermophilic less-acidophiles. In the second stage, the stabilized sludge (pH 4.5–5) from stage 1 was subjected to bioleaching in continuous stirred tank reactor (CSTR) under ambient conditions (37°C) using elemental sulfur as an energy source. Under mesophilic conditions acidophiles were expected to bring down pH from 5–4.5 to less than 2.5 required for efficient metal solubilization.

1 Materials and methods

1.1 Sludge collection

The undigested secondary activated sludge was procured from the bottom of a clarifier of a wastewater treatment plant in Delhi, the capital city of India (installed capacity > 100 million gallon/ day).

1.2 Inoculum preparation

The sulfur-oxidizing microorganisms indigenous to the sludge were used in inoculum preparation for ATAD operation. The activated sludge was initially fortified with 0.5% (*W/V*) elemental sulfur at 60°C and 180 r/min. When the pH of the sludge reduced from an initial value of 7 to 5, the culture was transferred to fresh sludge. This procedure was repeated three times so as to get an active inoculum (acclimatized sludge) for using it in the first stage ATAD operation. For second stage operation (metal bioleaching), the activated sludge was fortified with 0.5% elemental sulfur at 30°C and 180 r/min. When the pH of the sludge reduced from an initial value of 7 to 2, the culture was transferred to fresh sludge. This procedure was repeated three times so as to get an active inoculum for bioleaching of metals.

1.3 Characterization of sludge

The pH was determined immediately after sludge collection using Cyberscan 510 pH meter. The oxidation reduction potential (ORP) of the sludge was determined with standard ORP probe meter. For determination of total solids (TS), the sludge was kept in an oven at 105°C overnight. The total volatile solids (TVS) was measured by keeping the sludge in a muffle furnace at 550°C for 2 hr, whereas for suspended solids (SS) determination, the sludge was first filtered through a standard filter paper and then the residue remaining on the filter paper was kept in an oven at 105°C overnight. For volatile suspended solids (VSS) determination, the residue remained after filtering the sludge sample was kept in the muffle furnace at 550°C for 2 hr. The total nitrogen content of the sludge was examined using total kjeldahl nitrogen (TKN) method, whereas total phosphorus content was determined by stannous chloride method after acid digestion of the sludge sample. Total coliforms in the sludge were assayed on LES Endo agar (APHA, 2005). For total heavy metal determination, the sludge samples were subjected to di-acid digestion (HNO_3 + $HClO_4$) and the heavy metals in the digested liquid were determined using atomic absorption spectrophotometer (AAnalyst 200, PerkinElmer, USA). The physicochemical properties of the sewage sludge are shown in Table 1.

1.4 Autoheated thermophilic aerobic digestion (ATAD)

The batch ATAD experiment was performed with sludge having total solid of 40 g/L in a glass reactor having working volume of 7.5 L (Fig. 1). The reactor was well insulated with industrial glass wool followed by a layer of thermocol and aluminium foil. The flexible tubings (silicone rubber) used for connecting various units were also insulated in a similar manner. For efficient mixing, air was sparged at the bottom of the reactor at the rate of 1 vvm (volume of air/volume of reactor/min) through a diffuser having tiny holes of 1–2 mm diameter. A gas circulator was used to recycle the air from the top of the reactor and back to the reactor through the sparger. For optimum growth of microbes, the fresh air at the rate of 0.2 vvm was also provided. The sludge containing 10% (*V/V*) of the active inoculum and 0.5% (*W/V*) of elemental sulfur was fed into the reactor. A control experiment was carried out without the addition of sulfur and inoculum

Table 1 Physicochemical properties of sewage sludge

Parameter	Value
pH	7.3
Total solids (TS) (g/L)	40
Total volatile solids (TVS) (g/L)	31
Suspended solids (SS) (g/L)	38.8
Volatile suspended solids (VSS) (g/L)	30.2
Cu (mg/kg)	280
Ni (mg/kg)	130
Zn (mg/kg)	860
Mn (mg/kg)	120
Total kjeldahl nitrogen (%)	3.8
Total phosphorus (%)	1.4
Total coliform (CFU/100 mL)	109

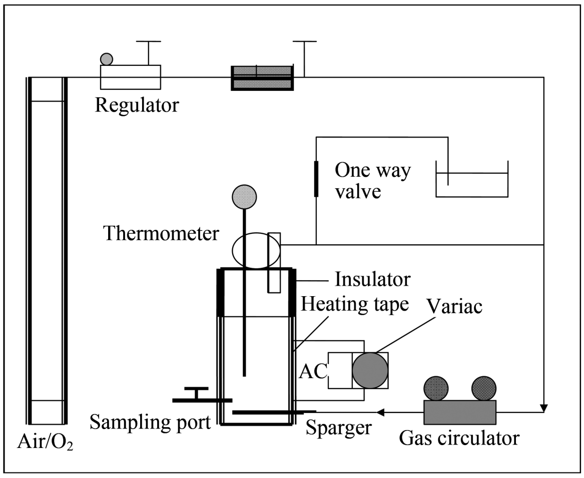

Fig. 1 Schematic diagram of the batch autoheated thermophilic aerobic digester.

to the sludge, which represents a conventional auto-heated thermophilic aerobic digestion. A heating coil was used to heat the digester and a temperature recorder was used to record the digester temperature.

In each experiment, electrical power input was initially selected to obtain a fixed 55°C. The ATAD experiment including control was conducted at this temperature. During the process, the reactor temperature increased from 55 to 60°C due to the heat produced by the biological oxidation of the sludge. Since, aerobic degradation of organics is an exothermic process, the heat will be continuously released and given a sufficiently solids concentration in the sludge, the reactor temperature will increased from 55 to 60°C.

1.5 Bioleaching experiments

The digested sludge (initial pH 4.6) from stage 1 was transferred to a feeding tank, kept for sufficient time to bring down the temperature at ambient level and thoroughly mixed with a stirrer. A CSTR (working volume 1.5 L) was initially fed with 10% (*V/V*) of active inoculum and 0.5% (*W/V*) elemental sulfur. The air was provided at 0.5 vvm for efficient mixing and maintaining sufficient oxygen level in the reactor and pH was monitored with time. As soon as the pH of the CSTR decreased to 2 or less, the sludge containing 5 g/L of sulfur was fed from the feeding tank to the CSTR through a peristaltic pump (Fig. 2). The CSTR was operated under ambient condition (37°C) at different mean hydraulic retention times (HRT) of 12, 24 and 36 hr to find out the optimum retention time for solubilization of heavy metals. The samples were taken regularly at every 6 hr and were analyzed for heavy metals content.

2 Results and discussion

2.1 Autoheated thermophilic aerobic sludge digestion

2.1.1 Change in pH and ORP

The change in pH with time during control and ATAD is shown in Fig. 3a. In the control without the addition of substrate and inoculum, pH increased from an initial value of 7.3 to 8 within 24 hr, due to the cell lysis at high temperature causing the release of alkaline matter. In ATAD process, in the presence of elemental sulfur and inoculum, pH decreased sharply to 5.4 in first 8 hr then decreased gradually and finally reached 4.6 in 24 hr. This decrease in pH from 7.3 to 4.6 indicates a better acclimatization and rapid growth of thermophilic sulfur-oxidizing microorganisms capable of oxidizing elemental sulfur.

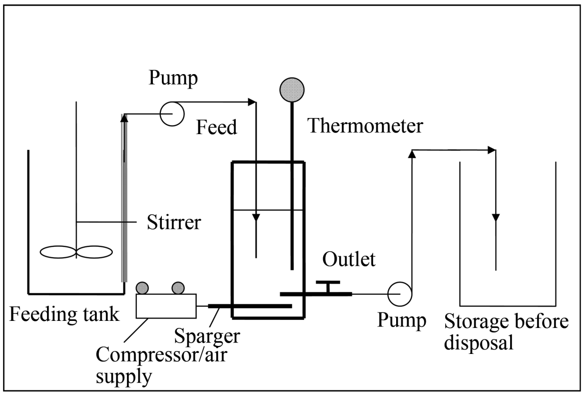

Fig. 2 Schematic diagram of the continuous stirred tank reactor (CSTR).

The change in ORP with time is shown in Fig. 3b. In the control, the ORP reached only 118 mV from an initial value of –79 mV, whereas in the ATAD, the ORP increased to a maximum of 192 mV from an initial value of –79 mV within 24 hr. The increase in ORP in ATAD was due to the oxidation of elemental sulfur by thermophilic sulfur-oxidizing microorganisms and is an indicator for the growth of *Acidithiobacilli* in the sludge. The above results show that indigenous thermophilic sulfur-oxidizing microorganisms were capable in oxidizing the sulfur leading to a decrease in pH and an increase in ORP of the sludge during ATAD.

2.1.2 Change in temperature profile

The temperature profiles of control and ATAD experiments are shown in Fig. 4. In the control, the temperature started increasing from 55°C and reached a maximum of 63°C in 16 hr and then remained constant up to 24 hr. However, temperature never exceeded 63°C in the control. This is due to the fact that when temperature reaches 63°C, any further increases in temperature will start inhibiting the aerobic digestion process. After 24 hr, temperature started decreasing from 63°C and reached 55°C. It was observed that in the control, autoheated conditions (55–60°C) could be maintained up to 96 hr (data not shown). Although, the desired temperature range (55–60°C) could be maintained for 96 hr, an increase in pH up to 8 in the control (Fig. 3a) would require a longer time and pre-acidification of the sludge for the second stage bioleaching process.

In the ATAD process containing inoculum and elemental sulfur, the temperature increased to a maximum of 62°C in 8 hr and then decreased to 55°C in 24 hr. After 24 hr the desired temperature range (55–60°C) in the ATAD could not be maintained. This is due to the lowering of pH in

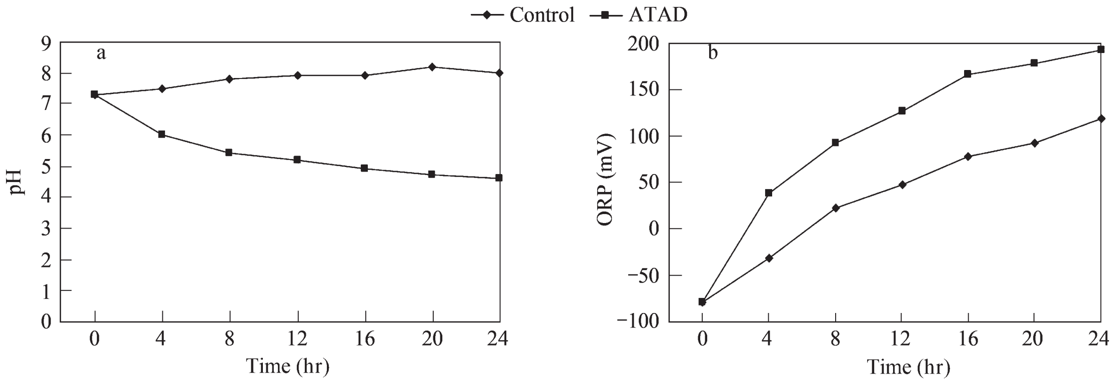

Fig. 3 Changes in pH (a) and ORP (b) with time in control and autoheated thermophilic aerobic sludge digestion (ATAD) process.

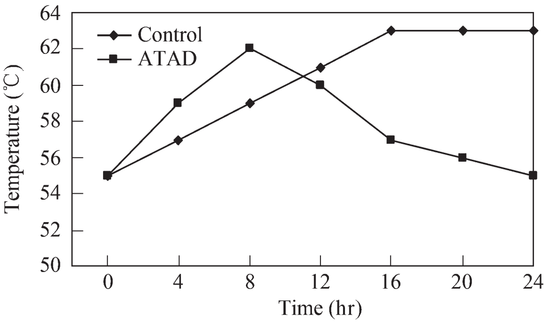

Fig. 4 Change in temperature during control and ATAD.

the reactor from 7.3 to 4.6 (Fig. 3a). The lowering of pH in the reactor was due to the growth of less-acidophilic thermophile, which inhibits aerobic digestion process. It is well known fact that aerobic digestion is favoured at near neutral pH of the sludge and even a small decrease in pH will inhibit the aerobic digestion, thus shortening the duration of heat released during the ATAD.

In the control and ATAD, the temperature of the reactor increased according to the magnitude of initial volatile suspended solids available in the sludge. Once the digestible solids were exhausted, temperature started decreasing and finally reduced to 55°C. It was reported that the maximum VSS degradation took place in the temperature range 55–60°C (Nosrati et al., 2007).

2.1.3 Reduction in sludge solids

As can be seen from Fig. 5a, in 24 hr, the reduction in TS, TVS, SS and VSS was 21%, 27%, 27% and 33% respectively in ATAD process. The reduction in solids was quite rapid, when temperature of the reactor was around 60°C. Almost 19% SS and 24% VSS were removed from the reactor in the first 12 hr. After this, temperature began to fall in the reactor, resulting in a slow reduction of solid within next 12 hr. However, the temperature range 55–60°C was enough to reduce 33% VSS, which was almost comparable to the result obtained for control reactor (34% reduction in VSS) during this period (Fig. 5b). As can be seen from the Fig. 4, the rate of increase in the temperature and duration for which reactor cam maintain the temperature range 55–60°C was lower in the ATAD, whereas almost similar reduction in sludge solids was achieved for ATAD and control (Fig. 5a). This can be explained on the basis of the pH profile of the ATAD (Fig. 3) in which, after 24 hr, the pH of the ATAD reactor reduced to 4.6. This decrease in pH led to the destruction of heterotrophic population resulting in a higher removal of VSS. The lowering of pH in ATAD also inhibits aerobic digestion process resulting in a small duration of heat produced during the process. In the control (Fig. 5b), the reduction in TS, TVS, SS and VSS was 23%, 28%, 28% and 34%, respectively, within 24 hr digestion which was almost similar to what was achieved in the ATAD. However, the positive aspect of the ATAD is the considerable reduction in solids as well as lowering of pH, which will reduce the time and pre-acidification for bioleaching in the second stage.

The results also show that the rate of VSS reduction (33%) during ATAD was higher in the present study

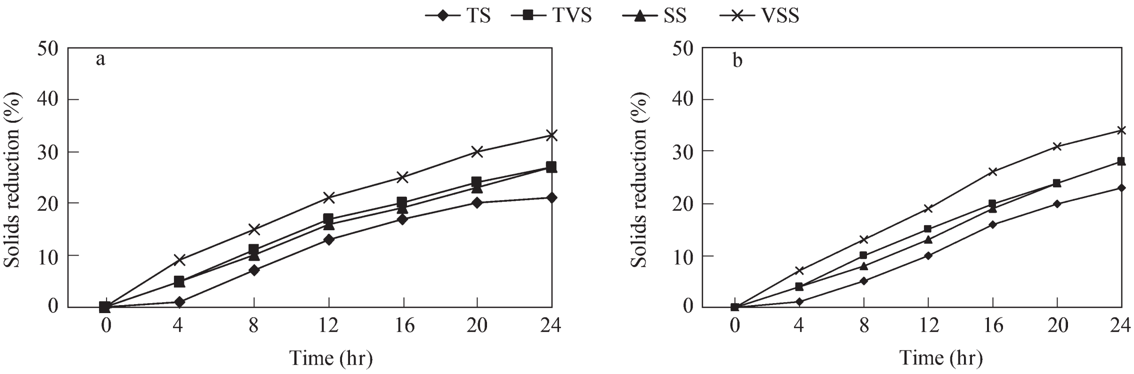

Fig. 5 Reduction of sludge solids in ATAD process (a) and in control (b).

compared to the previous study on thermophilic bioleaching at 53°C, where a maximum of 58% VSS reduction was achieved in fifteen days of thermophilic bioleaching (Shooner and Tyagi, 1996).

2.1.4 Destruction of pathogen

Table 1 shows the concentration of total coliforms in the sludge before digestion. In the present study, the total coliform count in the sludge before digestion was 109 cfu/100 mL. During aerobic digestion, the temperature in both the ATAD and control reactors was well above 55°C for 24 hr (Fig. 4). This has led to the destruction of total coliform, resulting in complete removal of indicator microorganisms in both the reactors. After 24 hr of digestion, the total coliform count was below the detection limit of 103 cfu/100 mL in both the reactors. It is well known that thermophilic aerobic digestion is efficient in removal of indicator bacteria (Kabrick and Jewell, 1982).

2.1.5 Removal of heavy metals during ATAD

As can be seen from Fig. 3, during ATAD, the pH decreased from an initial value of 7.3 to 4.6. This has led to the solubilization of 15% Cu, 11% Ni, 21% Zn and 16% Mn from the sludge. The significant solubilization during ATAD can be attributed due to the low pH coupled with high temperature, which breaks the sludge matrix thus releasing the metals into the liquid portion of the sludge. However, the digested sludge still contained significant amount of heavy metals which needed to be solubilized further. For this purpose, the sludge was subjected to continuous bioleaching in second stage of operation. The heavy metals content of the digested sludge is shown in Table 2.

2.1.6 Bioleaching of heavy metals in continuous stirred tank reactor

The operating conditions of each CSTR are given in Table 3. It is apparent from Table 3 that at higher HRTs, the pH in the reactor decreased, whereas the value of ORP increased. At a HRT of 12 hr, the pH of the CSTR was 2.3 under steady state condition. Further, increase in HRT to 24 hr and 36 hr resulted in pH decrease to 1.9 and 1.6, respectively. The average ORP for the CSTR operated at HRT of 12 hr was 480 mV, whereas ORP values were 512 mV at 24 hr and 534 mV at 36 hr. It is quite apparent that high ORP and low pH provides optimum conditions for metal solubilization.

Table 2 Characteristics of sludge from ATAD process

Parameter	Value
pH	4.6
TS (g/L)	31.6
Cu (mg/kg)	238
Ni (mg/kg)	115.7
Zn (mg/kg)	679.4
Mn (mg/kg)	100.8

The solubilization of Cu at different HRTs ranging from 12–36 hr is shown in Fig. 8. About 48% of residual Cu present in the digested sludge from stage 1 was solubilized in the second stage at an HRT of 12 hr. At HRT of 24 hr, significant solubilization was observed, which increased up to 72%. Further, increase in HRT to 36 hr increased the solubilization up to 81%. The increased Cu solubilization at higher HRT can be attributed to the longer extreme acidic conditions coupled with highly oxidizing conditions achieved at higher HRT. It has been reported that besides pH, ORP also plays an important role in solubilization of Cu.

To solubilize Cu, ORP of the medium should be more than 250 mV (Hayes and Theis, 1978). In the present study, the ORP in the reactor operated at an HRT of 12 hr was more than 450 mV. Further, the solubilization of Cu was found to be strongly correlated with ORP (r^2 = 0.977) and pH (r^2 = –0.969) of the sludge. The length of acidification is also an important parameter, which determines the solubilization of Cu. The higher solubilization of Cu at higher HRT also confirmed the above findings. The variance for different HRTs was also quite high (r^2 = 0.81) indicating that higher HRT favours the solubilization of Cu.

As shown in Fig. 6 among all the metals, Zn was present in maximum concentration (860 mg/kg). Even, at shortest HRT of 12 hr, more than 70% Zn was solubilized. At an increased HRT of 24 hr, 80% solubilization was achieved, which increased up to 92% for the reactor operated at mean HRT of 36 hr. The higher solubilization of Zn is due to the low pH and high ORP in all the reactors. According to Hayes and Theis (1978), to solubilize Cu below pH 5, the ORP of the medium should be greater than 250 mV, whereas at the same pH, the ORP requirement for Zn solubilization is –100 mV. In the present study, the pH

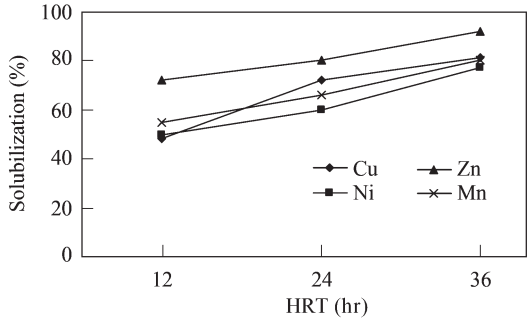

Fig. 6 Heavy metals solubilization at different hydraulic retention times (HRTs).

Table 3 Operating conditions in the CSTR

Hydraulic retention time (hr)	TS of feed sludge (g/L)	pH of feed	Reactor volume (L)	Mean ORP of the feed sludge (mV)	Final pH in the reactors	Final ORP in the reactors (mV)
12	31.6	4.6	1.5	192	2.3	480
24	31.6	4.6	1.5	192	1.9	512
36	31.6	4.6	1.5	192	1.6	534

was 2.3, 1.9 and 1.6 respectively in the reactors operated at mean HRT of 12, 24 and 36 hr. The ORP values were also quite high and were over 475 mV for all the three reactors. The solubilization of Zn also showed high correlation with pH (r^2 = –0.961) and ORP (r^2 = 0.951) suggesting that these two parameters were the major factors determining the solubilization of Zn in leaching medium.

Nickel was present in lower concentration compared to Cu and Zn. The solubilization of Ni also followed the same trend as that of Cu and at an HRT of 12 hr, 50% Ni was solubilized which increased gradually to 60% at an HRT of 24 hr. An HRT beyond 24 hr increased solubilization of Ni (77%). This can be explained on the basis of final pH (less than 2) achieved in the reactors operated at HRT of 24 and 36 hr. The solubilization of Ni was found to be strongly correlated with pH (r^2 = –0.947) and ORP (r^2 = 0.936).

Among all studied metals, Mn was present in the lowest concentration (100 mg/kg) in the sludge. The higher solubilization of Mn was achieved at higher HRT. At an HRT of 12 hr, 55% Mn was solubilized which increased up to 66% at an HRT of 24 hr and reached a maximum of 80% at an HRT of 36 hr.

In the present study, about 81% Cu, 92% Zn, 77% Ni and 80% Mn were solubilized in the second stage reactor at an HRT of 36 hr. The experiments beyond 36 hr HRT were not performed as the optimum conditions for metal removal such as low pH (1.6) and high ORP (534 mV) were already achieved. Further increase in HRT beyond 36 hr did not seem to bring down the pH further as the growth of microorganisms involve in bioleaching is inhibited at such a low pH. An increase in HRT beyond 36 hr will also add to the economy of the process. Moreover, under extreme low pH conditions, the conditioning and dewatering of the sludge become difficult. Keeping in mind the economy as well as the solubilization efficiency of the metals, an HRT of 24 hr appears to be a better option.

Besides metal solubilization, a marginal reduction in sludge solids (2%–4% in TS, 3%–6% VS and 4%–6% VSS) was observed in the reactors operated at mean HRTs of 12–36 hr. Therefore, the reduction in TS in both the stages was 23%–25%, whereas for the VS was 30%–33% operating at mean HRT of 12–36 hr. The total VSS reduction for the reactor operated at an HRT of 24 hr was 39%. The increased reduction in sludge solids (3%–6% for VS and 4%–6% for VSS) during bioleaching can be attributed to the acidic conditions developed in the reactor, which results in the destruction of heterotrophic population leading to high solids reduction.

The present study demonstrated that using a two stage process (ATAD and metal bioleaching), a total of 39% VSS reduction and removal of 76% Cu, 79.5% Ni, 78.2% Mn and 84.2% Zn could be removed within 50 hr. In an earlier study conducted by the present authors in batch reactors using elemental sulfur, it took sixteen days to achieve almost similar solubilization of the metals (Pathak et al., 2008). Furthermore, in the present study a higher solubilization (72% Cu, 80% Zn and 60% Ni) was achieved even at an HRT of 24 hr as compared to the reported values obtained in CSTR with pre-acidified sludge (initial pH 4) using *At. ferrooxidans*. In the above study, an HRT of three days (without recycling) was necessary to solubilize 65% Cu, 44% Zn and 23% Ni (Couillard and Mercier, 1991).

3 Conclusions

The results of the present study strongly indicated that it is possible to operate a two-stage reactor system which can address to the problem of sludge stabilization and metal bioleaching with high level of energy and time conservation. In the ATAD, after 24 hr of incubation time, 21% TS, 27% VS, 27% SS and 33% VSS were removed from the sludge. Further, the digested sludge from ATAD (stage 1) was free of pathogen and its pH was less than 5 (4.5–5). The digested sludge can be subjected to bioleaching without pre-acidification and an HRT of 24 hr was found to be sufficient to remove 72% Cu, 80% Zn and 60% Ni and 66% Mn from the sludge. The higher VSS reduction in the sludge also suggested that the digested sludge satisfies the class A norms (38% VSS reduction) set for the sludge disposal by USEPA.

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