

Leaching of phosphorus from incinerated sewage sludge ash by means of acid extraction followed by adsorption on orange waste gel

Biplob Kumar Biswas, Katsutoshi Inoue*, Hiroyuki Harada, Keisuke Ohto, Hidetaka Kawakita

Department of Applied Chemistry, Saga University, Honjo 1, Saga 840-8502, Japan. E-mail: bipshi2001@yahoo.co.uk

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Abstract

Ashes from sewage sludge incineration have a high phosphorus content, approximately 8% (W/W), which indicates a potential resource of the limiting nutrient. Incineration of sewage sludge with subsequent recovery of phosphorus is a relatively new sludge treatment technique. In this article, the leaching of phosphorus by using sulfuric acid as well as hydrochloric acid by means of several batch experiments was presented. At the same time a selective recovery of phosphorus by adsorption was also discussed. The effects of acid concentration, temperature and time on extraction were studied. The phosphorus leaching increased with the increase in acid concentration and temperature. Kinetic studies showed that the complete leaching of phosphorus took place in less than 4 h. Selective adsorption of phosphorus by using orange waste gel provided a hint for recovery of this natural resource, which eventually could meet the ever-increasing requirement for phosphorus. The overall results indicated that the incinerated sewage sludge ash can be treated with acid to efficiently recover phosphorus and thus can be considered a potentially renewable source of phosphorus.

Key words: sewage sludge ash; phosphorus; metals; acid extraction; recovery

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Introduction

Phosphorus is a limited non-renewable resource, which is indispensable as an essential nutrient for the growth of organisms in most ecosystems, and can not be replaced by other elements. It is a very important element for many industries as well. The demand for phosphorus fertilizer alone increased with the increase in the world's population from 9×10^6 to 40×10^6 metric tons between 1960 and 2000 and was expected to increase further to 20×10^6 metric tons by 2030 (Tilman *et al.*, 2001). The commercial products of phosphorus including fertilizer are principally produced from phosphate rock. However, there have been some alarming reports that deposits of high-grade phosphate ores are likely to be depleted in the next few decades (Franz, 2008; Runge-Metzger, 1995; Yoshida and Galinada, 2002). Therefore, it is very important to develop a sustainable method to recycle and conserve the phosphorus used in society to compensate for such global exhaustion of phosphate ores and to cope with a quite limited supply of phosphate in future, which can eventually lead to conserve phosphorus resource. Phosphorus can be recycled from any phosphorus-rich residues such as phosphorus-rich ash, sewage sludge and agricultural residue (Pettersson *et al.*, 2008a). However, in decision-making processes about materials management, recycling costs need to be compared with disposal costs (Inyang *et al.*, 2003).

Sewage sludge is one of the end-products of the physical, chemical and biological operations employed in modern wastewater treatment systems. The upgrading of treatment plants results in increased volumes of biological or chemical sludge, which may be more difficult to handle than a simple primary sludge. In addition, sludge volumes increase annually and more chemical sludge is produced with high metal and phosphate contents, there is the possibility to loss such elements associated with disposal (Tateda *et al.*, 1997). One option for sludge handling may be the digestion of the sewage sludge utilizing its energy content to produce biogas. However, the volume of the residual material remains almost the same as that of the original sludge. Moreover, this system requires additional processes to separate phosphorus which remains in the residue after digestion. Because of these considerations, incineration has grown in popularity as a final disposal process for sludge. Solid sludge incineration of municipal solid waste, which results in approximately 70% and 90% reduction in mass and volume respectively (Kosson *et al.*, 1996), is a component of integrated waste management in many countries. However, after incineration, the inorganic constituents (phosphorus and metals) are left in the ash (Scott, 1980).

One of the recovery methods is to use the sewage sludge as a fertilizer directly on soil/land. A decade ago there was a great interest in spreading sludge on agricultural land due to the potential for recycling valuable components

* Corresponding author. E-mail: inoue@elechem.chem.saga-u.ac.jp

such as organic matter, phosphorus and nitrogen which are essential nutrients for plants (Fuentes *et al.*, 2004). In the developing countries almost all of the sludge is disposed off in open fields due to the shortage of appropriate disposal facilities. But, in the long run this system may cause a serious problem with increasing trace elements in soil, groundwater and surface water because generally phosphorus (Ekholm and Krogerus, 1998) and heavy metal levels are higher in sludge than in soil. Recently, there has been an increased concern of the heavy metal concentration in sewage sludge (Lue-Hing *et al.*, 1998) due to its non-biodegradability, toxicity and consequent persistence (Dutta, 2002).

In recent years, effective removal methods for phosphorus as well as metals from sludge have been extensively studied (Fytianos and Charantoni, 1998; Matsuo, 1996; Pettersson *et al.*, 2008a, 2008b; Wong and Henry, 1988). These methods include a bioleaching process (Solisio *et al.*, 2002), an electrokinetic process (Alshawabkeh and Bricka, 2000), supercritical extraction (Smart *et al.*, 1997), chemical extraction, and so forth. Although bioleaching is thought to offer more advantages over acid treatment, the effectiveness of this process in natural conditions is yet to be tested. In addition, since bioleaching is an aerobic process, supply of enough oxygen is a critical parameter which has to be monitored very carefully along with the temperature. Moreover, nitrogen and phosphorus are found to remain unchanged after bacterial leaching (Wong and Henry, 1984). The electroreaccumulation process, on the other hand, is based on electrokinetic phenomena that occur when the sludge is electrically charged with direct current by means of several arrays of electrodes (Acar and Alshawabkeh, 1993). Quite understandably the high energy consumption of this system proved it to be an unattractive technology for extraction.

Chemical extraction is a promising method for separating contaminants from sewage sludge ash by using chemicals to reduce the amount of elements present in the ash (Babel and Dacera, 2006) and to remedy soil contaminated with heavy metals (Paff *et al.*, 2004). The extractants include inorganic acids (H_2SO_4 , HCl , HNO_3), organic acids (citric and oxalic acids), inorganic chemicals (e.g., ferric chloride) and chelating agents (e.g., ethylenediaminetetraacetic acid). Although quantitative removal is possible with inorganic chemicals such as $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, qualitative extraction (100%) is not attained readily because a long reaction time is required. However, inorganic acids provide better extraction efficiencies than other extractants. Hong *et al.* (2005) studied phosphorus recovery from waste-activated sludge incinerator ash and found that 2 mol/L HCl could leach phosphorus out of the ash but it could extract no more than 69% of the major metals content (Ca, Al and Fe). However, for phosphorus recovery, they recommended solvent extraction using organic solvents, which was thought to incur extra cost for the successful application of this system. Adsorption is one of the processes that can uptake phosphorus from dilute solutions and thus can be considered to be suitable for the recovery of dilute concentration of phosphorus. By utiliz-

ing proper synthesis route, available agricultural byproduct can be transformed into an efficient adsorption gel at very cheap cost. Therefore, from the viewpoint of cost effective phosphorus recovery, adsorption technique can be employed in line with extraction. In our previous work (Biswas *et al.*, 2008), we developed an adsorption gel using orange waste precursor, modified it by loading Zr(IV) and tested the adsorption capacity for phosphorus under various conditions. The present study was undertaken to obtain laboratory data for an acid (low concentration) extraction process to investigate the potential for phosphorus recovery from incinerated sewage sludge ash (ISSA). Sulfuric acid as well as hydrochloric acid was used at different concentrations and temperatures to establish the optimum recovery conditions for phosphorus. The leaching of other major elements such as Ca, Fe, Al in ISSA was also studied to better understand the extraction behavior of major elements other than phosphorus. At the same time, an adsorbent prepared from orange waste precursor was used to recover phosphorus selectively from the leachate.

1 Materials and methods

1.1 Characterization of incinerated sewage sludge ash (ISSA)

1.1.1 Analysis of the ISSA sample

The sample of ISSA was collected from Kumamoto Nambu Sewage Treatment Center, Japan and stored in desiccators at room temperature until use. The sludge pH was measured in deionized water at a sludge/solution (m/V) ratio of 25:1 for 48 h stirring at room temperature and was found to be 7.24. The average water content of the as-received ISSA was determined by heating a measured amount of sample to 105°C for 24 h (Cheeseman *et al.*, 2003). The surface area of the ISSA sample was measured according to BET method using a Belsorp 18PLUS (Bel. Inc., Japan).

1.1.2 Particle size distribution

The particle size distribution of the ISSA was measured by using a particle size separator consisting of four sieves, which separated the bulk sample into five particle size ranges (< 53 , $53\text{--}75$, $75\text{--}150$, $150\text{--}300$, and $> 300\ \mu\text{m}$). The sieves were placed one on top of another in such a way that the size of the opening decreased from top to bottom. The ISSA sample (10 g) was placed on the top sieve and shaken to achieve separation into fractions.

1.1.3 Microstructural analysis

The crystalline phases present in the ISSA were identified by X-ray diffractograms (XRD-610D, Shimadzu, Japan) at 30 mA and 40 kV. The drive axis was 2θ ranging from 1.5 to 80° while the continuous scan speed was $2^\circ/\text{min}$. This allows identification of major crystalline phases but does not give any information on amorphous or glassy materials present in the sludge ash. However, the $53\text{--}75\ \mu\text{m}$ and $75\text{--}150\ \mu\text{m}$ particle size fractions of the ISSA were mixed together to make the sample for XRD analysis. The surface morphology was observed by a

digital microscope (VH-5000, Kyence, Japan) as well as by a scanning electron microscope (S-3000N, Hitachi, Japan).

1.1.4 Total dissolution of ISSA

Total dissolution experiments were individually carried out by using concentrated HCl, H₂SO₄, HNO₃ and aqua regia at their boiling temperatures. The insoluble residue was double baked until totally dissolved. The digestion liquid was diluted with deionized water and filtered using a 1- μ m ashless filter paper. Finally, the metal as well as the phosphorus content of the filtrate was determined using a Shimadzu model ICPS-8100 ICP/AES spectrometer.

1.2 Adsorption material

The preparation of adsorption material from orange waste has been described in detail elsewhere (Biswas *et al.*, 2008). About 100 g orange waste was taken along with 8 g Ca(OH)₂ and crushed to make small particles. The mixture was then transferred into a beaker and, after adding a substantial amount of water, the suspension was stirred for 24 h at about 200 r/min at room temperature to facilitate saponification. The pH of this suspension was maintained constant at around 12.5 by adding sodium hydroxide solution. After stirring, the suspension was repeatedly washed with deionized water until neutral pH by means of decantation and finally filtered to obtain a wet gel, which was dried in a convection oven for about 48 h at 70°C to produce a dry gel. The adsorption gel thus produced is termed as saponified orange waste (SOW) gel, which was further modified by loading with Zr(IV). Approximately 3 g SOW gel was equilibrated with 500 mL of 0.1 mol/L zirconium solution at pH 2.11 for 24 h. The suspension was then filtered and washed until neutral pH, followed by drying in vacuum. The dried gel was finally sieved to produce a particle size fraction of between 75 and 150 μ m.

1.3 Leaching tests

The leaching test procedure described by Nagib and Inoue (2000) was followed in this study. The ISSA sample was leached using different types of leach liquors such as H₂SO₄, HCl and NaOH at varying concentrations (0.01–2.0 mol/L). All reagents used during the experiments were of analytical grade. A liquid/solid (L/S) ratio of 20 to 250 mL/g was used in all cases. Leach liquor was added to a measured amount of the ISSA sample (200 mg) in a conical flask, which was then placed in a thermostated shaker (Thomas thermostatic shaking incubator AT24R) at constant temperature and shaken at 140 r/min for 4 h to allow for complete reaction. After this time, the slurry was immediately filtered through a 1- μ m ashless filter paper and the filtrate was analyzed for phosphorus and metals by ICP/AES spectrometry. The factors affecting the leaching process such as acid or alkaline concentration, time, temperature etc. were thoroughly examined. However, all measurements were carried out in triplicates and average values were taken. Before leaching, all glassware was treated in a solution of 10% HNO₃ for 24 h and washed with deionized water.

1.4 Leaching kinetics

The leaching rate of phosphorus and other metals from ISSA was studied as follows: approximately 500 mL of 0.05 mol/L H₂SO₄ were added to 3.33 g ISSA in an Erlenmeyer flask to make a L/S ratio of 150 mL/g. The flask was put into a water bath to maintain a constant temperature. The whole slurry was stirred with a magnetic stirrer. Samples were taken out at defined time intervals from the suspension and filtered with a 1- μ m ashless filter paper. The concentrations of phosphorus and other metals in the filtrate were determined.

1.5 Adsorption of phosphorus from acid leachate

Zr(IV)-loaded SOW gel was used to adsorb phosphorus from the acid leachate, which contains not only phosphorus but also other metal ions at different solid/liquid ratio. To carry out this batch experiment different weight of Zr(IV)-loaded SOW gels were put in conical flasks in each of which 10 mL of leachate was poured and equilibrated at 30°C for 24 h. The concentration of phosphorus and other metal ions before and after equilibration were measured to determine the extent of adsorption.

2 Results and discussion

2.1 Characteristics of ISSA sample

The residual water content present in the as-received ISSA sample was found to be 1.1 wt.%. It was shown from the analysis of digital microphotograph (Fig. 1a) as well as scanning electron micrograph (Fig. 1b) that ISSA sample consisted of aggregated small particles with very irregular shapes such as flakes, many spheres and sintered agglomerated particles. Such shapes are quite common in incinerated ash. The particle size distribution of ISSA sample is shown in Fig. 2, from which it was found that 35% and 34% of the sample belong to 75–150 μ m and 53–75 μ m fractions, respectively, while the amount of the largest particle size fraction (> 300 μ m) was very small. The specific surface area of ISSA was measured to be 4.7 m²/g.

The elemental composition of the ISSA is shown in Table 1, which demonstrates that the sample contains P, Ca, Al and Fe as the major elements. From the viewpoint of the rich phosphorus content, ISSA appeared to be an attractive resource if phosphorus could be recovered. Therefore, our main objective was to recover phosphorus from ISSA through acid extraction processes using optimum conditions.

However, instrumental analysis of the ISSA sample was carried out to ascertain the presence of major elements found earlier from the ICP analysis after performing a total dissolution of the sludge ash. The X-ray diffractogram pattern for ISSA sample indicated that the major crystalline phases present in the ISSA were copper manganese oxide (Cu_{1.2}Mn_{1.8}O₄), iron phosphate (Fe₄(P₄O₁₂)₃), aluminum phosphate (Al(PO₃)₃), iron oxide or hematite (Fe₂O₃) and calcium oxide (CaO).

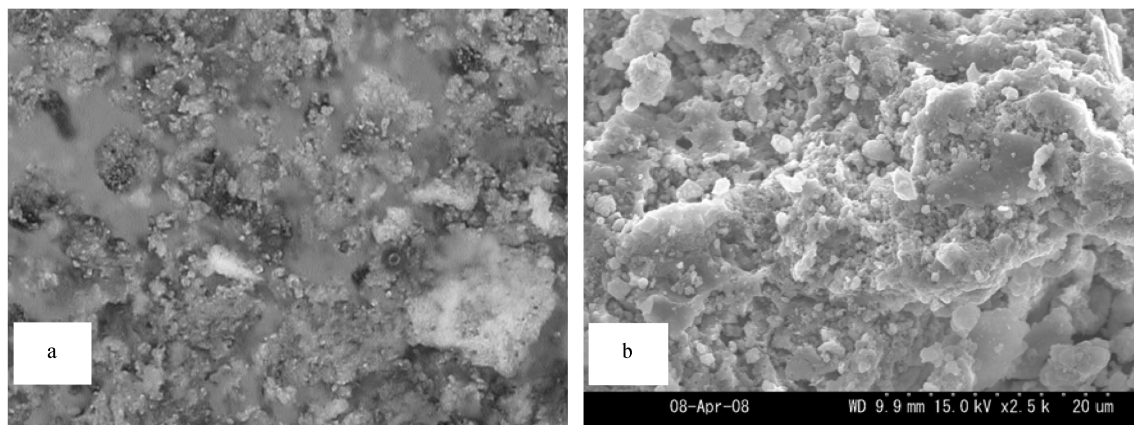


Fig. 1 Surface morphology of incinerated sewage sludge ash by digital microphotography (800 times magnification) (a) and scanning electron micrograph (2500 times) (b).

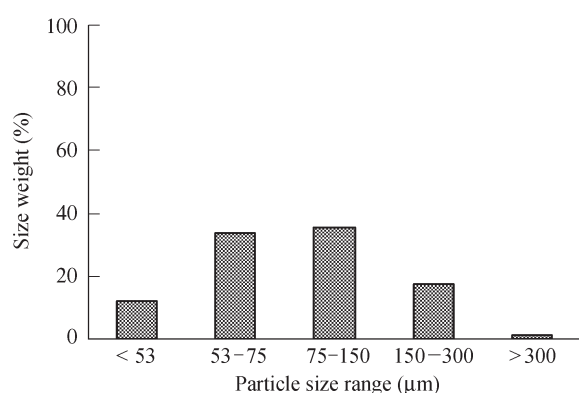


Fig. 2 Particle size distribution of ISSA.

Table 1 Elemental composition of the ISSA (mg/g)

Element	Dissolving reagent			
	Aqua regia	H ₂ SO ₄	HCl	HNO ₃
P	88.0	86.9	81.3	83.0
Ca	56.5	58.9	53.9	56.4
Al	60.4	33.0	34.7	41.6
Fe	52.1	17.9	46.8	11.3
Mg	19.3	11.8	15.8	16.1
Mn	1.22	0.748	1.10	1.04
Cu	0.715	0.498	0.569	0.508
Zn	0.763	0.447	1.03	0.583
Cd	0	0	0	0
Pd	0	0	0	0

2.2 ISSA sample leaching tests

To evaluate the leaching behavior of phosphorus and other metals such as Fe, Ca, Al, Mg and Mn, different acid leach liquors were investigated. The cost (Wzorek *et al.*, 2006) of 1 mol hydrogen ions required for an ash leaching solution under strongly acidic conditions increases in the series H₂SO₄, HCl, HNO₃, and H₃PO₄. Therefore, from an economical point of view, sulfuric acid and hydrochloric acid were used to extract phosphorus and other metal ions. It is quite well known that, if acid is added to sludge, the heavy metals present in the sludge are dissolved and enter into solution. Consequently, phosphorus also enters the solution due to the breakage of the metal-phosphorus bonds.

Sodium hydroxide was also employed for leaching; but it achieved only less than 40% leaching of phosphorus. A comparative extraction capabilities of H₂SO₄, HCl and NaOH are depicted in Fig. 3, which clearly shows that acid extraction is very effective for phosphorus release while extraction with NaOH is not suitable for this purpose. As a result, sodium hydroxide was not used in further extraction tests.

2.2.1 Sulfuric acid

Effects of time, acid concentration and temperature on ISSA leaching were examined (Figs. 4 and 5). The effect of sulfuric acid concentration on phosphorus leaching at different L/S ratios is depicted in Fig. 4, which shows that the leaching percentage of phosphorus increases with an increase in both the L/S ratio and acid concentration (0.01–0.1 mol/L). At concentrations of 0.2 mol/L and higher, the leaching of phosphorus is nearly 100% irrespective to the L/S ratio. Considering the cost-effective consumption of acid and achievement of complete leaching of phosphorus, 0.05 mol/L sulfuric acid at a L/S ratio of 150 mL/g is considered as the most suitable condition for leaching.

Figure 5 shows the leaching percentage of phosphorus and other metal ions from the ISSA at L/S ratio of 150 mL/g at varying sulfuric acid concentrations. This figure

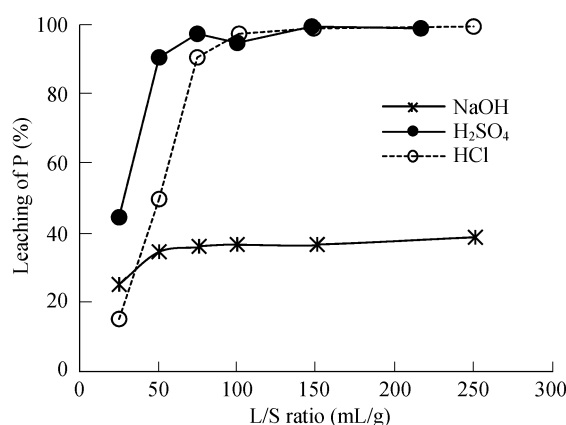


Fig. 3 Effect of H₂SO₄, HCl and NaOH on the leaching of phosphorus from ISSA. Conditions: concentration 0.1 mol/L (each); temp. 30°C; contact time 24 h.

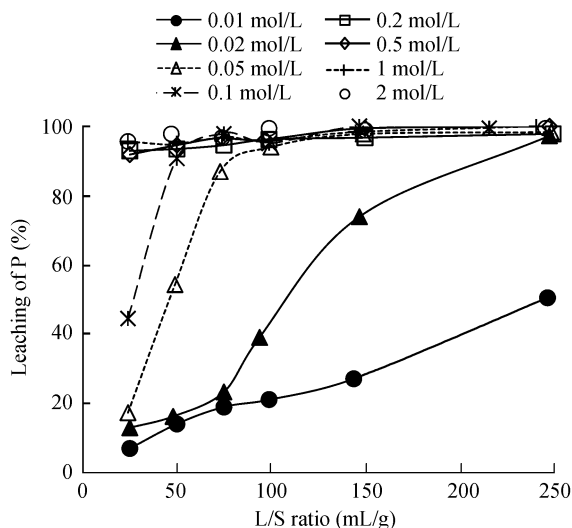


Fig. 4 Effect of H_2SO_4 concentration and L/S ratio on the leaching of phosphorus from ISSA. Conditions: temp. 30°C ; contact time 4 h.

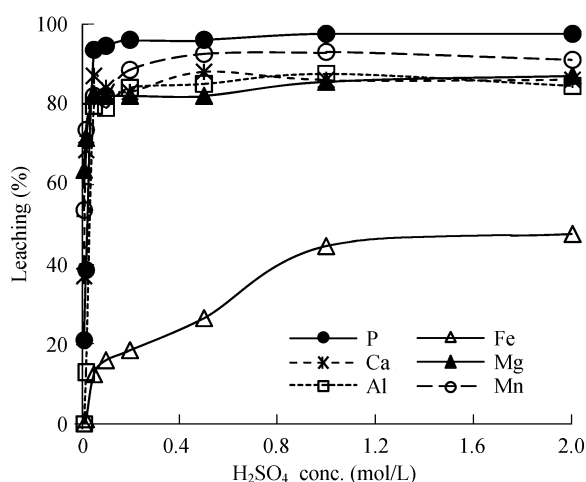


Fig. 5 Effect of H_2SO_4 concentration on phosphorus and metal leaching from ISSA. Conditions: L/S ratio 150 mL/g; temp. 30°C ; contact time 4 h.

shows that the solubility of phosphorus is higher than that of other metals which indicates that P exists in the ISSA as an acid soluble form. It also shows that the extraction of iron is much less than other metals, which may be attributable to the low solubility of the iron compound, because, during the incineration process, the iron might have been transformed to iron oxide (Stark *et al.*, 2006) which is not dissolved in acid. Takashi *et al.* (2001) reported that the acid-insoluble component consisted not only of SiO_2 but also of Fe_2O_3 , which seems to be the case in the present study. Figure 5 also shows that phosphorus is almost totally leached with increasing acid concentration while there is no change in the release of other metals, which may be due to the fact that the remaining fractions of those metals are present as nucleation particles in the center matrix of ISSA. A similar observation was also reported by Hong *et al.* (2005) for waste-activated sludge.

2.2.2 Hydrochloric acid

The effect of hydrochloric acid concentration on phosphorus leaching has been studied, which is shown in Fig. 6. Similar to the effect of sulfuric acid, this figure shows that the leaching percentage of phosphorus increases with increasing L/S ratio and hydrochloric acid concentration. At concentrations of 0.5 mol/L and higher, the leaching of phosphorus is nearly 100% irrespective to the L/S ratio. Taking consumption of acid into account, 0.1 mol/L HCl at a L/S ratio of 150 mL/g seems to be best suited condition for complete phosphorus leaching.

2.3 Kinetic study

Solubility and mass transfer rate are considered to be the most important factors which govern the rate of leaching kinetics from the solid phase to the liquid phase (Kosson *et al.*, 1996). In order to better understand the extraction behavior, the leaching percentage of phosphorus and other metal ions with sulfuric acid and hydrochloric acid has been plotted against shaking time in Figs. 7a and 7b, respectively, which show similar kinetic behavior. Figures 7a and 7b indicate that the leaching of each element increases with increasing leaching time and eventually reaches plateau region corresponding to each element. Almost all of the phosphorus was leached within less than 2 h while 90% of Ca and 65% of Al were leached by this time. However, 4 h was chosen for acid extraction of the ISSA for phosphorus leaching experiments to ensure complete extraction. The possible reason for the easy leaching of phosphorus is due to its presence as soluble salts in the ISSA, which has been confirmed by XRD. This conclusion has been well-supported by Hong *et al.* (2005) who reported that phosphorus would be present in the incinerator ash in the form of highly soluble salts.

2.4 Effect of temperature

Temperature was found to affect the leaching of elements from ISSA by using sulfuric acid as illustrated in Fig. 8, which shows the extraction percentage by using 0.05 mol/L H_2SO_4 as a function of temperature. The leaching percentage increases very smoothly with increasing

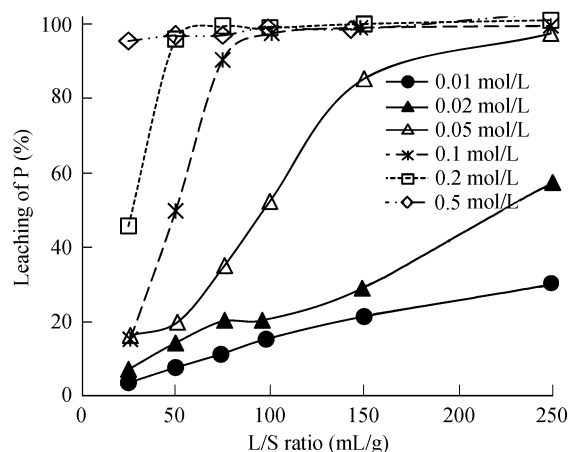


Fig. 6 Effect of HCl concentration and L/S ratio on the leaching of phosphorus from ISSA. Conditions: temp. 30°C ; contact time 4 h.

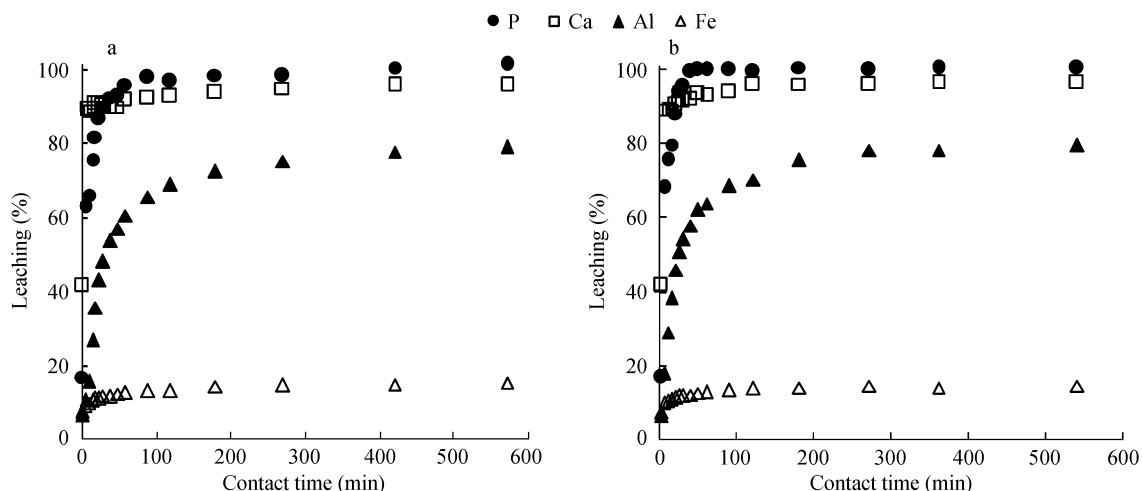


Fig. 7 Effect of shaking time on ISSA leaching using 0.05 mol/L H₂SO₄ (a) or 0.1 mol/L HCl (b). Conditions: L/S ratio 150 mL/g; temp. 30°C.

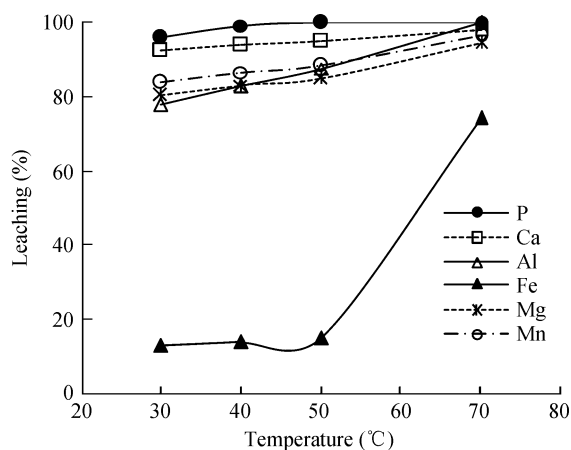


Fig. 8 Effect of temperature on phosphorus and metal leaching from ISSA using H₂SO₄. Conditions: L/S ratio 150 mL/g; H₂SO₄ concentration 0.05 mol/L; contact time 4 h.

temperature for P, Ca, Mg, Al and Cu. This result can be explained by the fact that an increase in temperature provides energy necessary for breakdown of the chemical bonds of the metals in the sludge ash (Naoum *et al.*, 2001). On the other hand, the leaching percentage for Fe remains constant (ca.15%) from 30 to 50°C but increases to 75% at 70°C, suggesting that iron compound in ISSA needs higher energy (in the form of either temperature or acid concentration) to be broken. However, 30°C was determined to be suitable for further leaching. The selection of this temperature, however, avoids high solubility of other metals such as Fe and Cu.

2.5 Selective recovery of phosphorus from acid leachate by adsorption

Adsorption is a useful tool for recovery of elements from solution. Zr(IV)-loaded SOW gel was employed to adsorb phosphorus from sulfuric as well as hydrochloric acid leachate. The leachates of pH 1.7–1.9 were directly used without any pH adjustment. Figure 9 shows that the percentage of phosphorus adsorption increases with the increase in adsorbent dose and reaches maximum at 100 irrespective to the leachate type. In the previous work

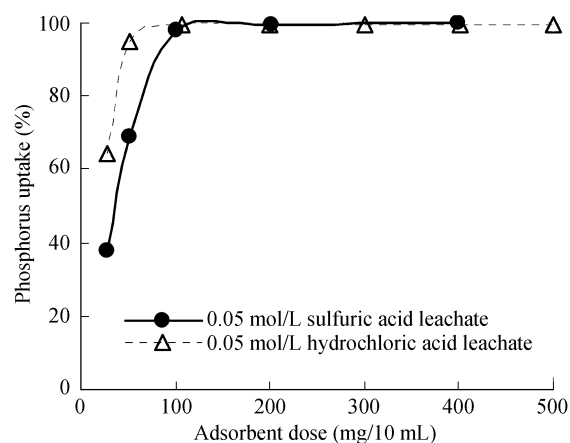


Fig. 9 Adsorption of phosphorus from H₂SO₄ and HCl leachate by using Zr(IV)-loaded SOW gel. Conditions: equilibration time 24 h; temp. 30°C.

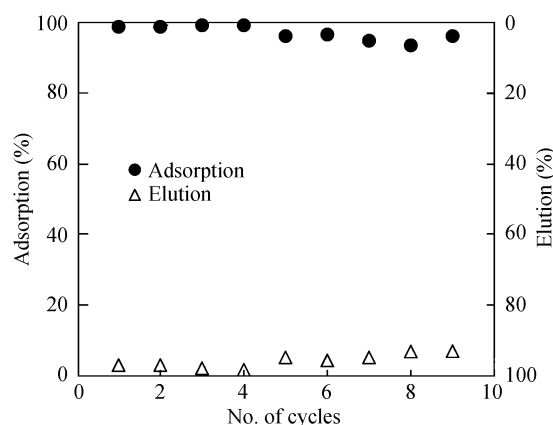


Fig. 10 Cycle test for the reutilization of Zr(IV)-loaded SOW gel. Conditions: phosphate concentration 38 mg/L; pH 3.24; eluting agent 0.2 mol/L NaOH.

(Biswas *et al.*, 2008), we found that phosphate is adsorbed onto the Zr(IV)-loaded SOW gel with the substitution of OH- and/or H₂O ligands, coordinated with the Zr(IV) ions at its hydrated condition, which has been further supported by the fact that the pH of the solution has been either increased or not changed at all after the adsorption of phosphate. However, in this experiment, the metal ions

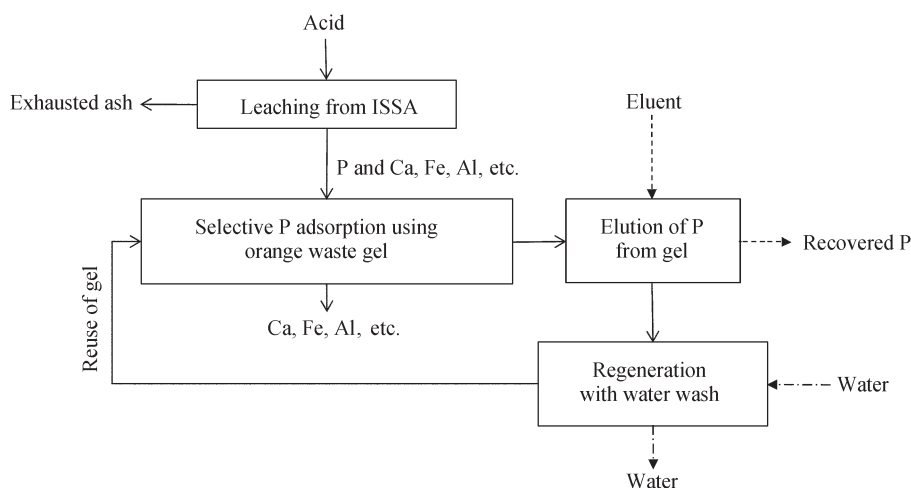


Fig. 11 Flowsheet of leaching of phosphorus and subsequent adsorption and elution followed by regeneration of adsorption.

such as Ca, Al, Fe etc. were not adsorbed because the cation adsorption sites of the gel are already occupied by Zr(IV) during its loading. In addition, the binding constant for Zr(IV) loading was large enough not to be leaked from the gel matrices. Thus, a selective adsorption of phosphorus could lead this system suitable for resource recycling and conservation. However, to confirm reutilization of the Zr(IV)-loaded SOW gel, cycle tests were carried out by using a solution containing 38 mg P/L at pH 3.24. This test was run up to nine adsorption-elution cycles and a good extent of adsorption as well as elution was achieved as shown in Fig. 10. Sodium hydroxide (0.2 mol/L) was used as an eluent since the authors found it to be efficient for elution of phosphorus (Biswas *et al.*, 2008). A full flow-sheet for leaching-adsorption-elution-regeneration is depicted in Fig. 11, which shows the whole process.

3 Conclusions

A series of extraction studies have been performed for the view of recovery of phosphorus from ISSA, which leads a step towards sustainable management and conservation of resources. The overall findings of this laboratory investigation confirmed a strong case for ISSA to be beneficially leached to recover phosphorus. This study indicated that phosphorus as well as other metal ions could be recovered from incinerated sewage sludge ash by using sulfuric acid or hydrochloric acid as the leach liquor. Quantitative leaching of phosphorus was achieved with an optimum condition as follows: 0.05 mol/L H_2SO_4 or 0.1 mol/L HCl at L/S ratio of 150 mL/g at 30°C for 4 h of shaking. Since the application of sewage sludge in agriculture is going to decline, phosphorus recycling from the sludge ash becomes more and more pertinent. Selective adsorption of phosphorus on Zr(IV)-loaded SOW gel after extraction from ISSA and recovery of this limiting nutrient have given an innovative finding for phosphorus recovery, which may provide valuable information for solving the problem of global shortage of phosphorus.

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